The Multicomponent Reaction of Imidazo[1,5-a]pyridine Carbenes with Aldehydes and Dimethyl Acetylenedicarboxylate or Allenoates: A Straightforward Approach to Fully Substituted Furans

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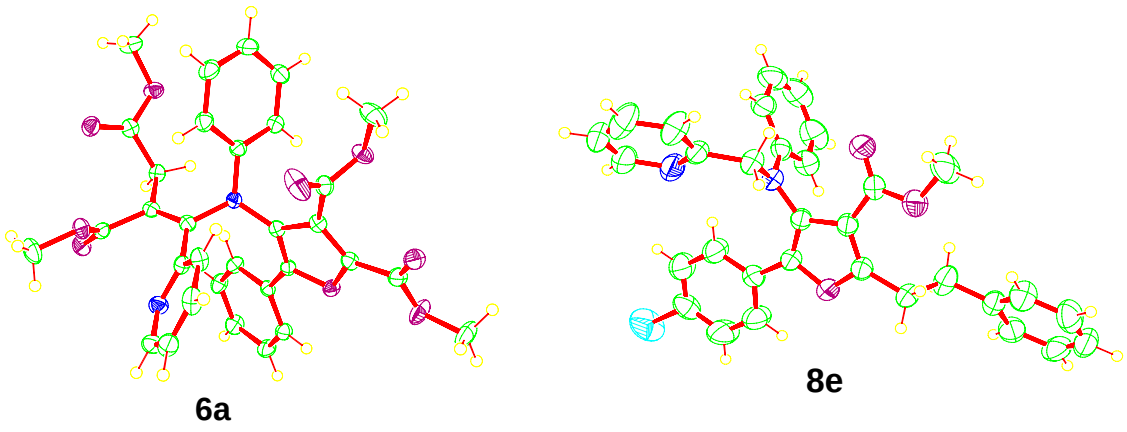
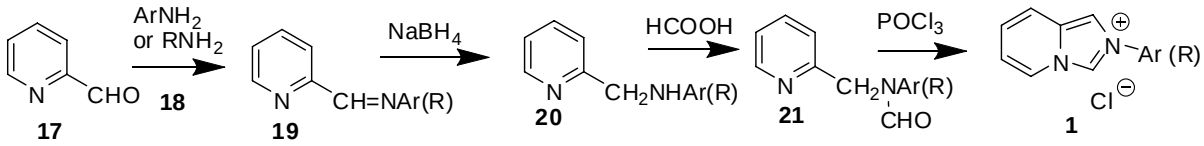


Figure S1. ORTEP drawing of X-ray structures of compounds **6a** and **8e**.



General procedure for the preparation of 2-aryl (alkyl)imidazo[1,5-*a*]pyridinium salts 1

At 0°C, amines **18** (0.1 mol) were added to the mixture of picolinaldehyde **17** (0.1 mol) and anhydrous $MgSO_4$ (5g) in dry diethyl ether (150 mL). The reaction mixture was stirred at room temperature for 8-12h. After removal of $MgSO_4$ and solvent, the imines **19** were obtained as yellow solids. The imines **19** were reduced by $NaBH_4$ (0.13 mol) in anhydrous ethanol (200 mL) at room temperature for 24h. After the evaporation of ethanol, the residue was added slowly to water (100 ml) and the aqueous solution was extracted with chlorform (100×5 mL). The extraction was dried and removed solvent to give amines **20** as yellow solids. The amines **20** were formylated by refluxing in anhydrous formic acid (60 mL) for 8h. After removal of formic acid under vacuum, the residue was added to water (20 mL) and was neutralized with solid Na_2CO_3. The aqueous solution was extracted with chlorform (100×5 mL), and the combined extraction was dried and evaporated to afforded amides **21** as white solids. The amides **21** reacted with $POCl_3$ (0.13 mol) in refluxing toluene (60 mL) for 3 days, and then toluene and the excess $POCl_3$ were removed under vacuum. The residue was chromatographed on a neutral Al_2O_3 column eluting with a mixture of ethanol and acetone (1:4) to give imidazo[1,5-*a*]pyridinium salts **1** in 27-86% total yields of four steps from picolinaldehyde **17**.

2-Phenylimidazo[1,5-*a*]pyridin-2-ium chloride 1a (Known compound)[1]: 86%, white solids, mp 96-97 °C; δ_H (400 MHz, $CDCl_3$) 12.0 (s, 1H), 9.35 (d, *J* = 7.1 Hz, 1H), 8.02 (s, 1H), 7.78 (d, *J* = 7.7 Hz, 2H), 7.64 (d, *J* = 9.4 Hz, 1H), 7.49-7.57 (m, 3H), 7.17 (dd, *J* = 9.3, 2.5 Hz, 1H), 7.01 (t, *J* = 6.9 Hz, 1H).

2-(4-Methoxyphenyl)imidazo[1,5-*a*]pyridin-2-ium chloride 1b (Known compound)[1]: 85%, white solids, mp 217-218 °C; ν_{max}/cm^{-1} 3060, 1655, 1608, 1593, 1545, 1519; δ_H (400 MHz, $CDCl_3$) 12.0 (s, 1H), 9.41 (d, *J* = 7.1 Hz, 1H), 7.91 (s, 1H), 7.76 (d, *J* = 8.9 Hz, 2H), 7.63 (d, *J* = 9.4 Hz, 1H), 7.23 (d, *J* = 9.2 Hz, 1H), 7.04-7.10 (m, 3H), 3.88 (s, 3H).

2-(4-Chlorophenyl)imidazo[1,5-*a*]pyridin-2-ium chloride 1c

[2-(4-Chlorophenyl)imidazo[1,5-*a*]pyridin-2-ium perchlorate is known[2]]: 69%, white solids, mp 244-245 °C; δ_H (400 MHz, $CDCl_3$) 12.2 (s, 1H), 9.33 (d, *J* = 7.0 Hz, 1H), 7.97 (s, 1H), 7.86 (d, *J* = 8.7 Hz, 2H), 7.66 (d, *J* = 9.9 Hz, 1H), 7.61 (d, *J* = 8.7 Hz, 2H), 7.28-7.31 (m, 1H), 7.11 (t, *J* = 6.9 Hz, 1H).

2-(4-(Trifluoromethyl)phenyl)imidazo[1,5-*a*]pyridin-2-ium chloride 1d: 27%, white solids, mp 256-257 ^{o}C; ν_{max}/cm^{-1} 3061, 1656, 1615, 1548; δ_H (400 MHz, $CDCl_3$) 12.1 (s, 1H), 9.18 (d, *J* = 7.1 Hz, 1H), 8.35 (s, 1H), 8.15 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.5 Hz, 2H), 7.74 (d, *J* = 9.4 Hz, 1H), 7.24 (dd, *J* = 9.4, 2.5 Hz, 1H), 7.10 (t, *J* = 6.9 Hz, 1H); δ_C (100 MHz, DMSO-d_6) 138.1, 130.7, 130.5, 130.2, 129.9, 129.8, 127.52, 127, 49, 127.45, 127.41, 126.5, 125.4, 124.9, 124.2, 123.8, 122.2, 118.3, 112.2; HRMS (TOF-ESI): 263.0790 (M^+); $C_{14}H_{10}F_3N_2^+$ required 263.0791.

2-Isopropylimidazo[1,5-*a*]pyridin-2-ium chloride 1e: 46%, 38-39 ^{o}C; ν_{max}/cm^{-1} 3123, 1655, 1626, 1544; δ_H (400 MHz, $CDCl_3$) 11.3 (s, 1H), 9.07 (d, *J* = 7.1 Hz, 1H), 8.29 (s, 1H), 7.76 (d, *J* = 9.4 Hz, 1H), 7.18 (dd, *J* = 9.2, 6.6 Hz, 1H), 7.01 (t, *J* = 6.7 Hz, 1H), 5.16 (quintet, *J* = 6.7 Hz, 1H), 1.76 (d, *J* = 6.7 Hz, 6H). δ_H (100 MHz, $CDCl_3$): 129.8, 125.8, 124.5, 118.14, 118.1, 117.1, 111.2, 54.3, 23.3; HRMS (TOF-ESI): 161.1072 (M^+); $C_{10}H_{13}N_2^+$ required 161.1073.

2-Benzylimidazo[1,5-*a*]pyridin-2-ium chloride 1e (Known compound)[3]: 75%, white solids, mp 44-45 ^{o}C; δ_H (400 MHz, $CDCl_3$) 11.4 (s, 1H), 8.94 (d, *J* = 7.0 Hz, 1H), 7.99 (d, *J* = 8.8 Hz, 1H), 7.58 (d, *J* = 9.2 Hz, 2H), 7.36 (d, *J* = 2.7 Hz, 2H), 7.13 (t, *J* = 9.2 Hz, 1H), 6.97 (t, *J* = 6.9 Hz, 1H), 5.87 (s, 2H).

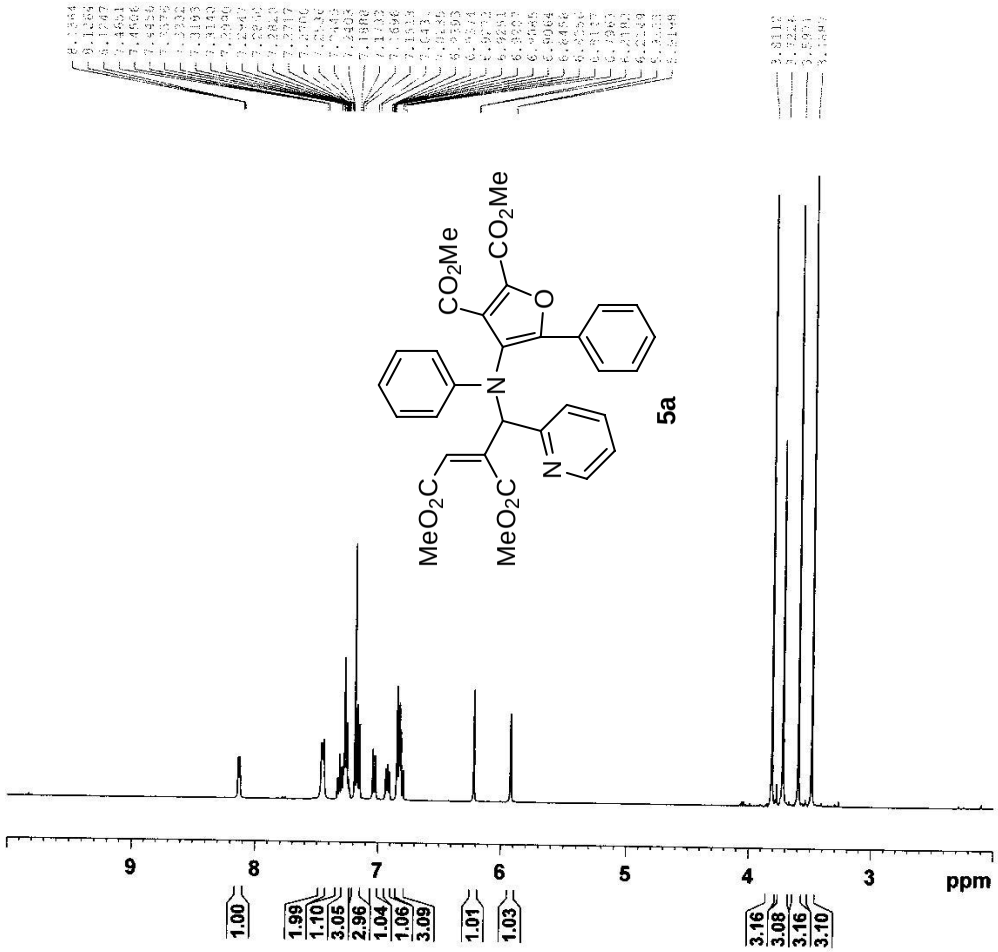
2-(4-Methylphenyl)imidazo[1,5-*a*]pyridin-2-ium chloride 1g

[2-(4-Methylphenyl)imidazo[1,5-*a*]pyridin-2-ium perchlorate is known[2]]: 81%, white solids, mp 217-218 ^{o}C; δ_H (400 MHz, $CDCl_3$) 11.9 (s, 1H), 9.40 (d, *J* = 7.1 Hz, 1H), 8.02 (s, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 9.4 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.23 (dd, *J* = 9.2, 6.9 Hz, 1H), 7.06 (t, *J* = 6.9 Hz, 1H); δ_C (100 MHz, DMSO-d_6) 140.3, 132.6, 130.6, 129.7, 125.7, 125.1, 124.2, 122.3, 118.2, 118.0, 112.0, 20.6.

1. Chien, C.-H.; Fujita, S.; Yamoto, S.; Hara, T.; Yamagata, T.; Watanabe, M.; Mashima, K. *Dalton Trans.* **2008**, 916-923.

2. (a) Mishra, D.; Naskar, S.; Adhikary, B.; Butcher, R. J.; Chattopadhyay, S. K. *Polyhedron* **2005**, *24*, 201-208. (b) Chattopadhyay, S. K.; Mitra, K.; Biswas, S.; Naskar, S.; Mishra, D.; Adhikary, B.; Harrison, R. G.; Cannon, J. F. *Transition Met. Chem.* **2004**, *29*, 1-6.

3. Alcarazo, M.; Roseblade, S. J.; Cowley, A. R.; Fernandez, R.; Brown, J. M.; Lassaletta, J. M. *J. Am. Chem. Soc*. **2005**, *127*, 3290-3291.



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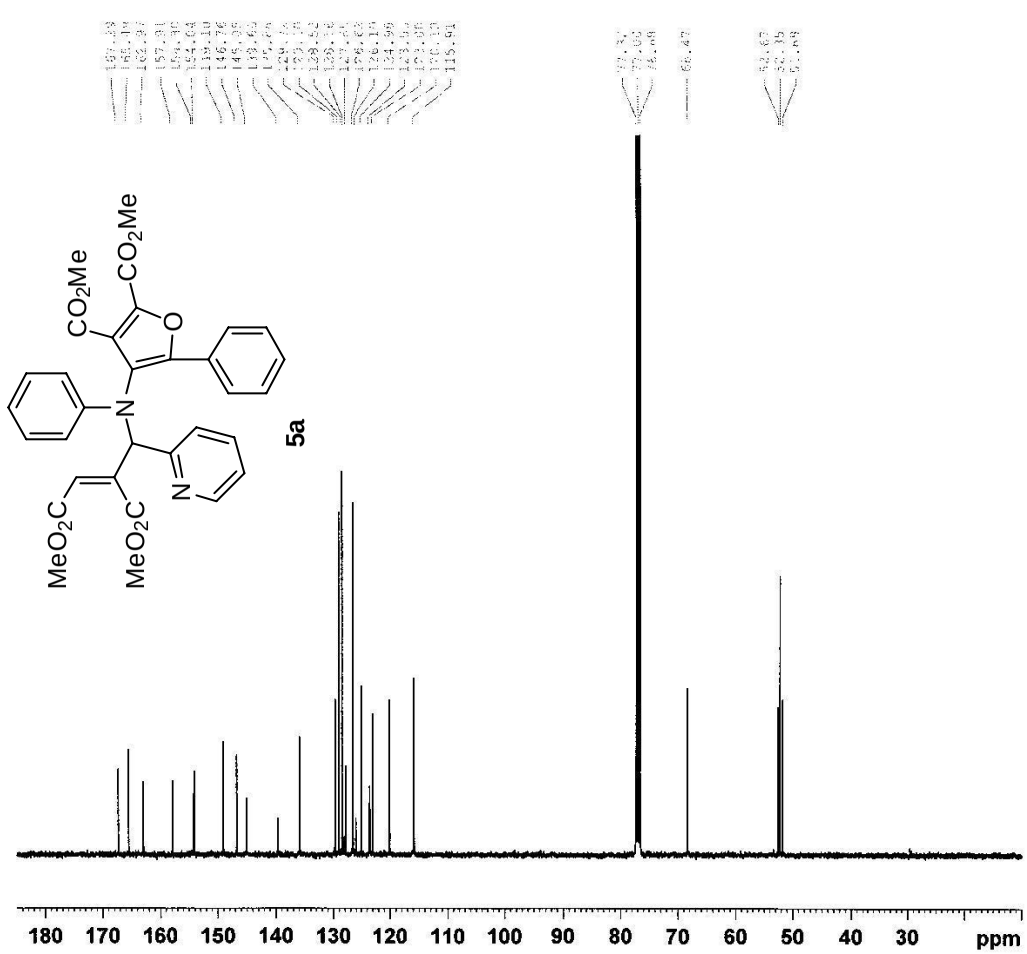

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PL1                -2.00 dB
SFO1         100.6328888 MHz

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NUC2                 1H
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PL2                -1.00 dB
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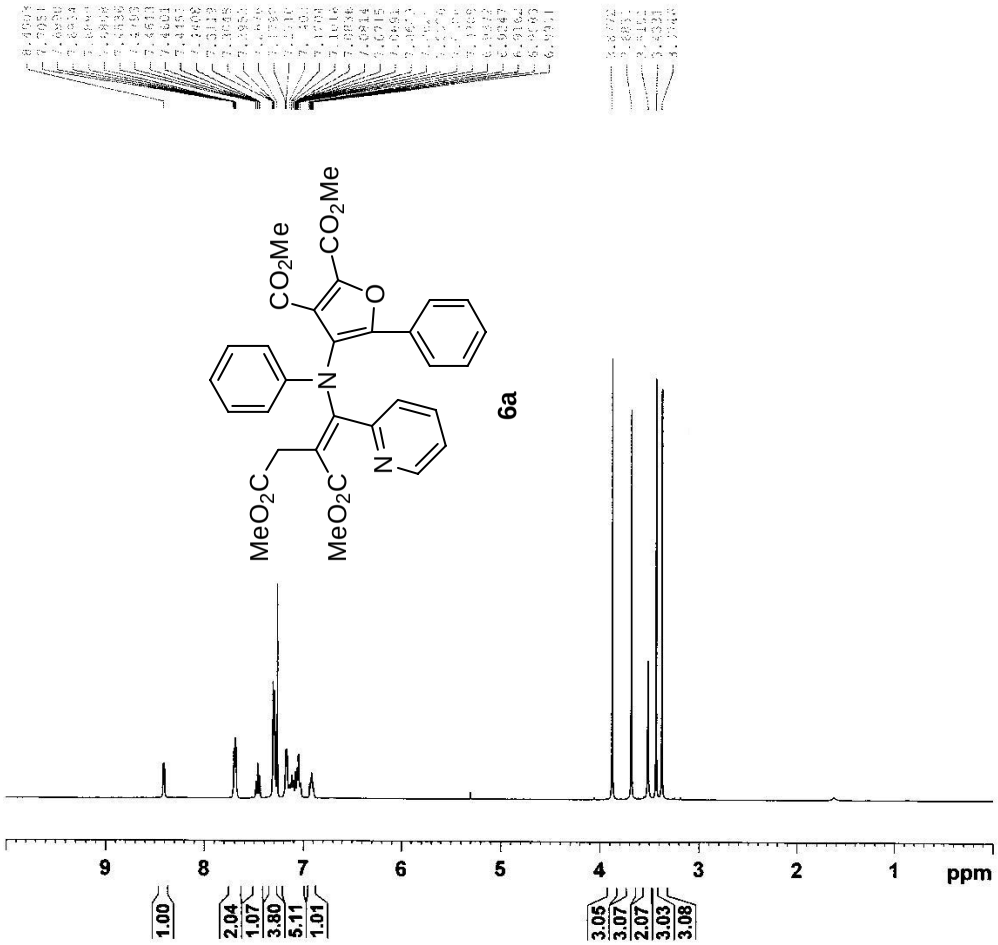

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TE                     297.0 K
D1                  1.00000000 sec
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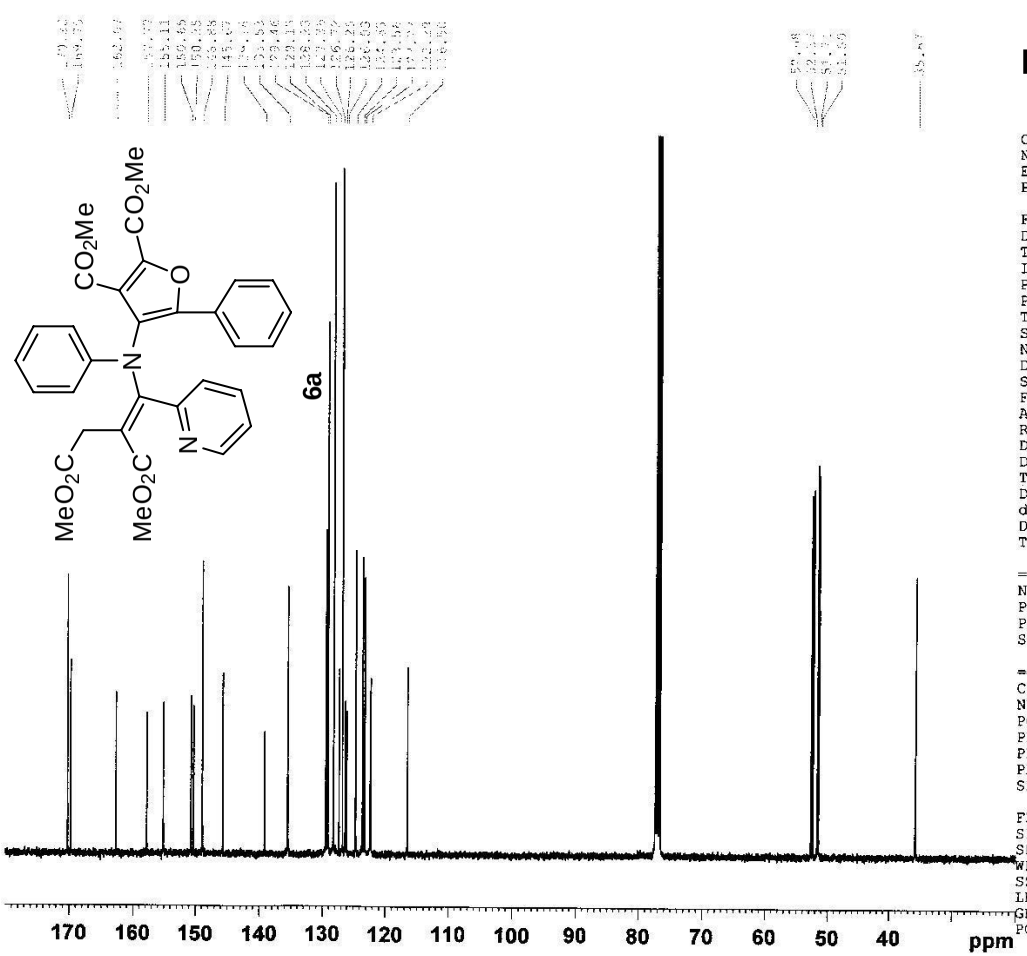

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AQ             1.3631988 sec
RG                   203
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DE               6.50 usec
TE              300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
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TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                8.40 usec
PL1              -2.00 dB
SFO1        100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
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PL13             14.46 dB
PL2              -1.00 dB
SFO2        400.1716007 MHz

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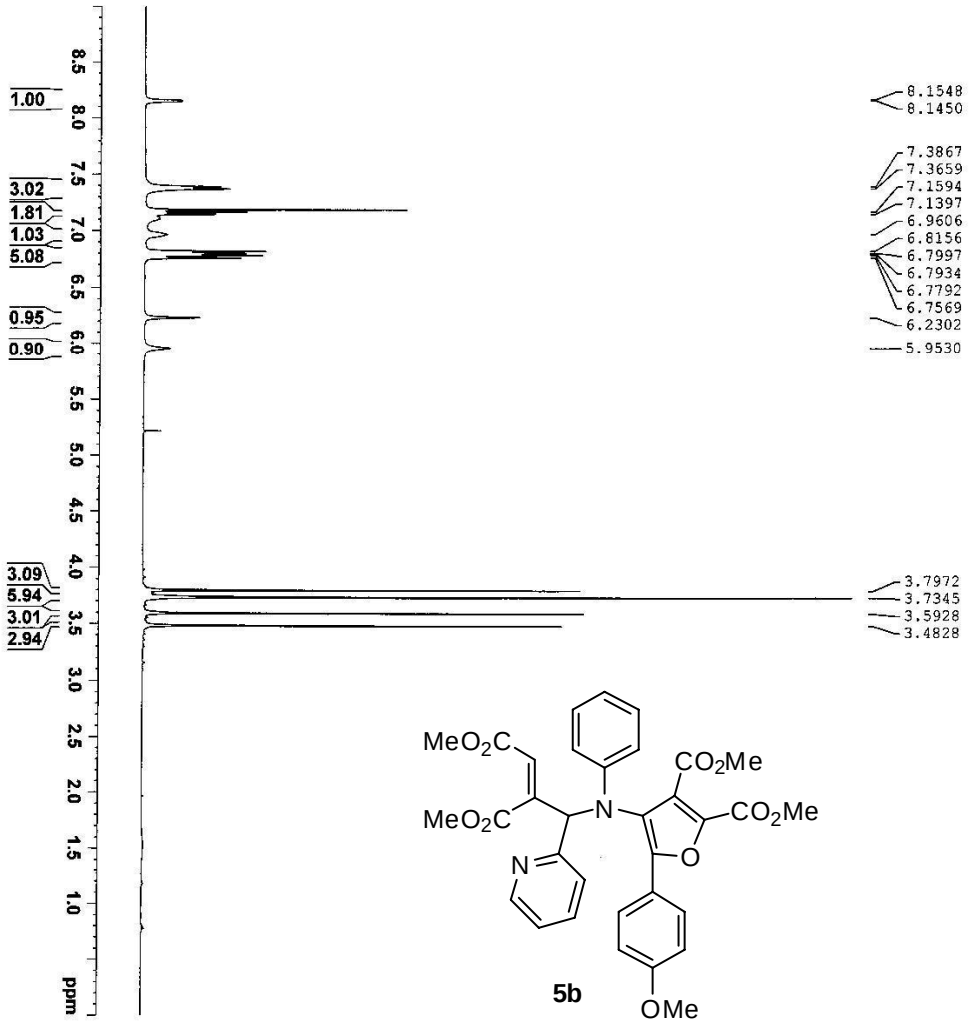

5b

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NS                        16
DS                         2
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FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       203
DW                    60.800 usec
DE                     6.50 usec
TE                    296.2 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.80 usec
PL1                    -1.00 dB
PLIW               13.18669796 W
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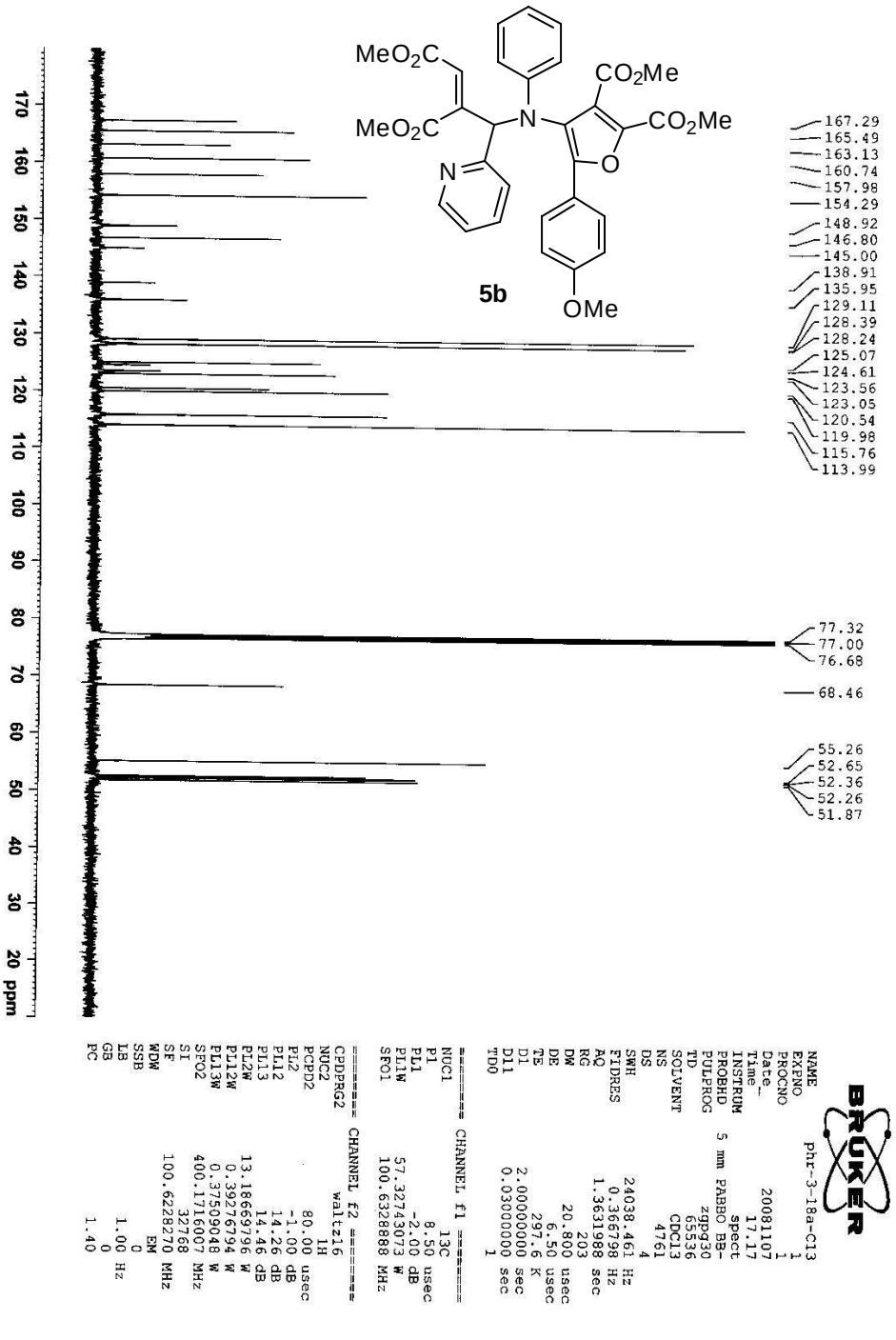
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RG 203
DW 20.800 usec
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TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
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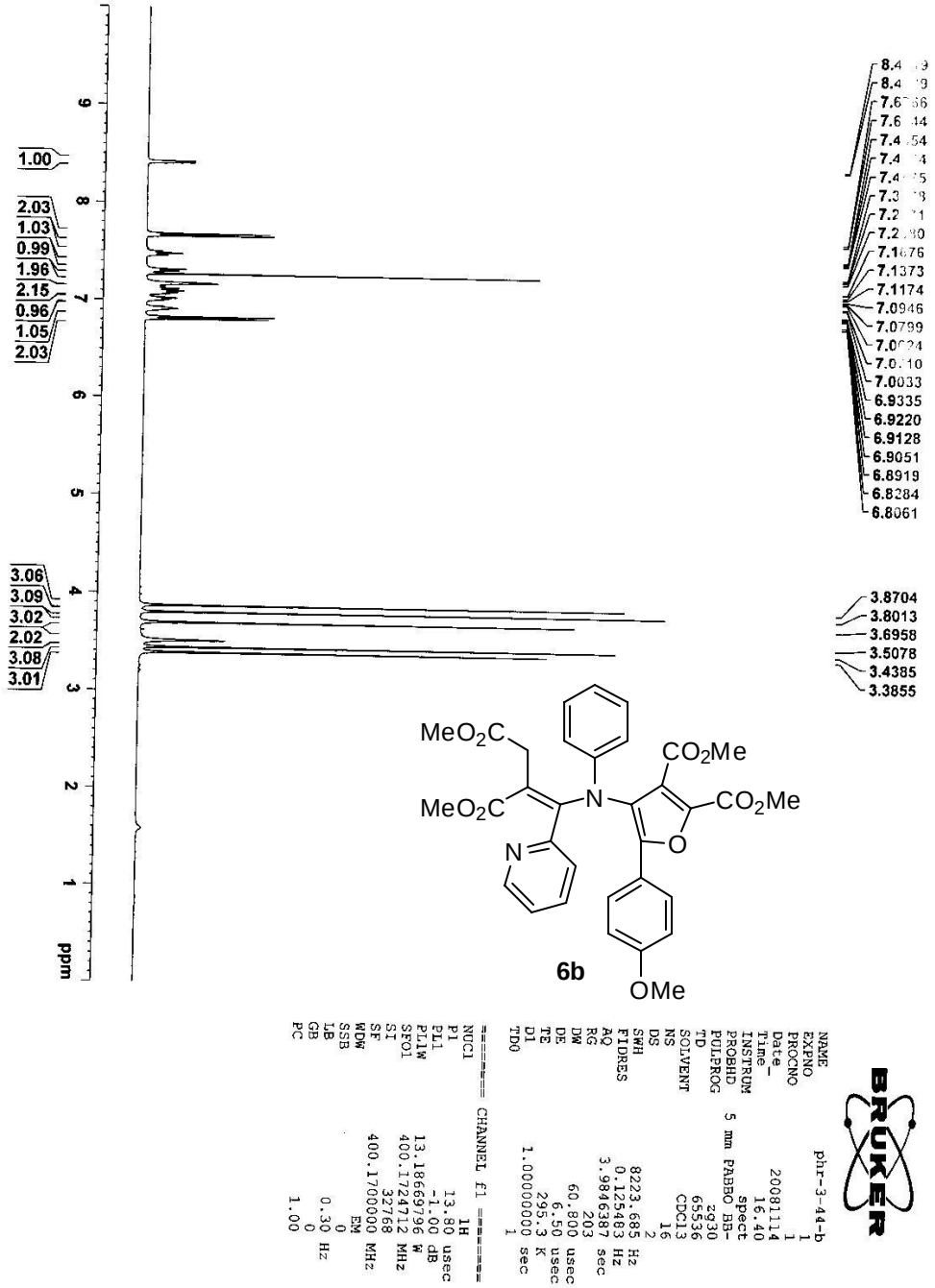


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SOLVENT                 CDCl3
NS                         16
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FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                        203
DW                   60.800 usec
DE                    6.50 usec
TE                    295.3 K
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TD0                           1

======== CHANNEL f1 ========
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SFO1               400.1724712 MHz
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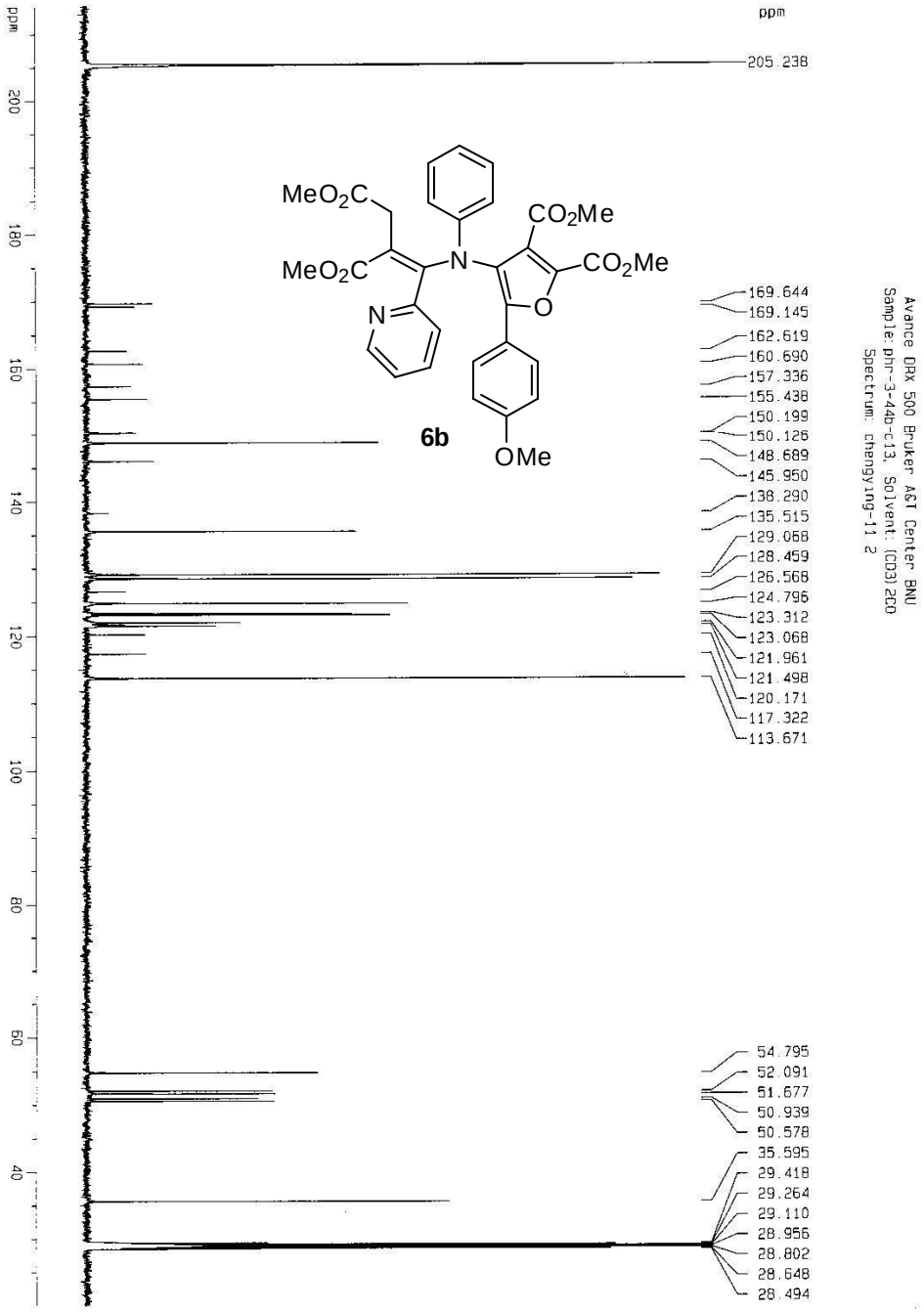
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6b
Avance DRX 500 Bruker AST Center BNU
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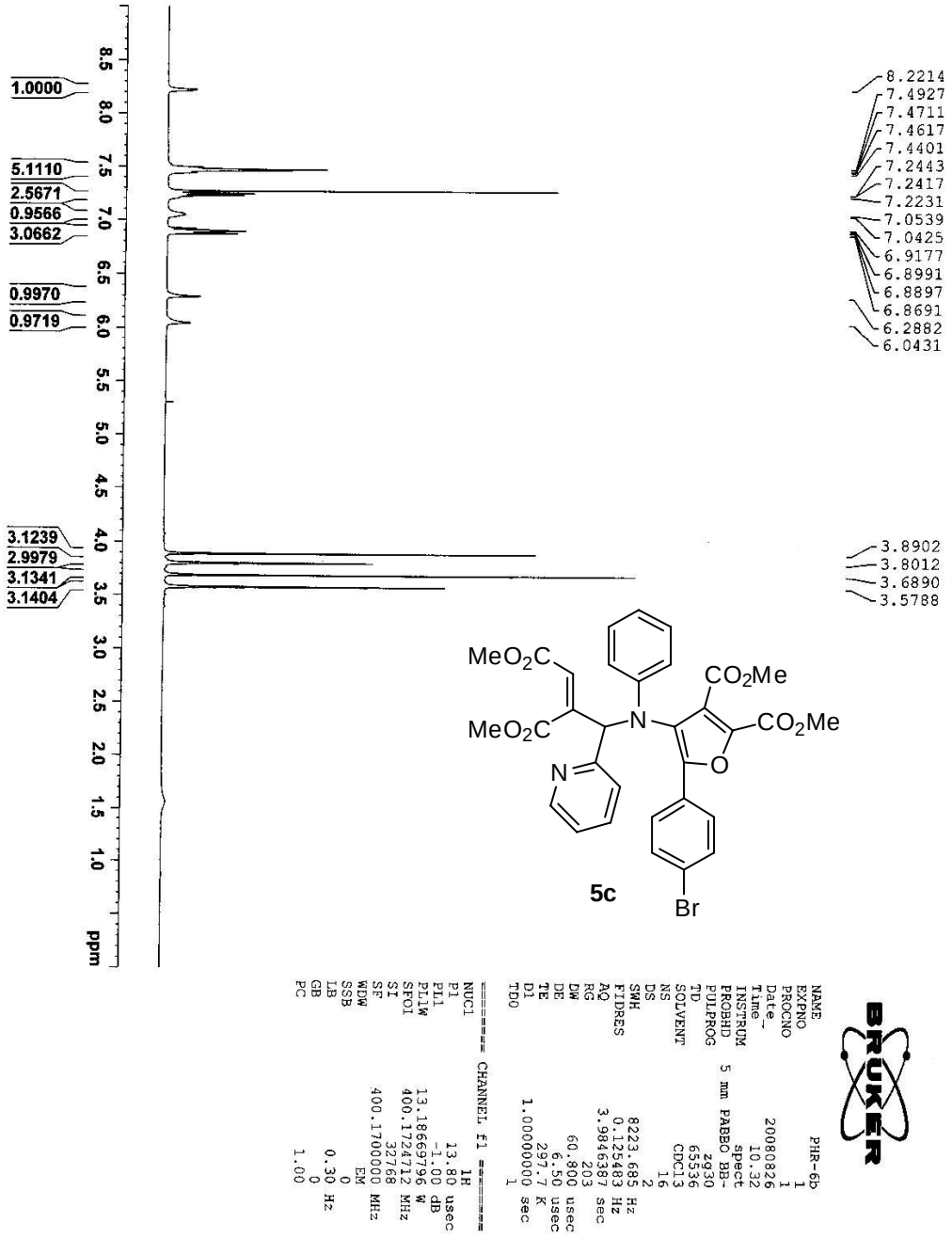
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NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
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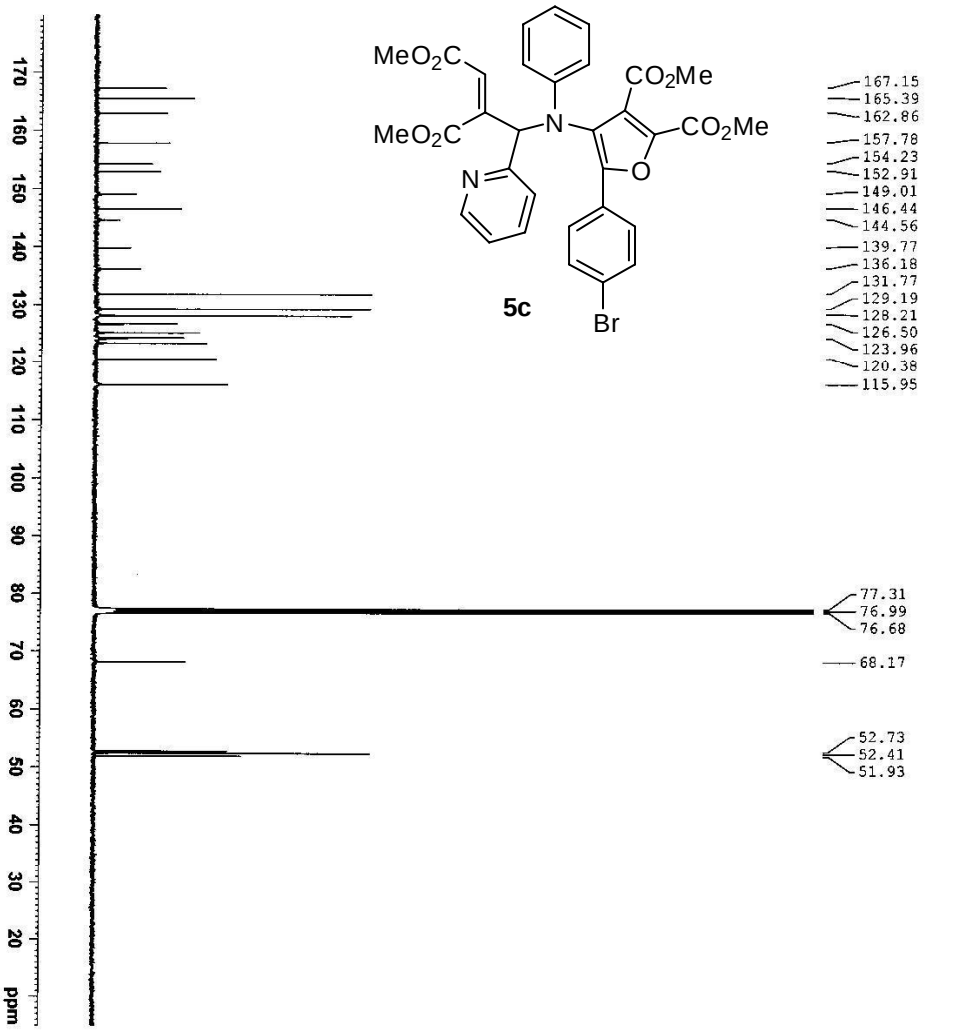


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FIDRES                  0.366798 Hz
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RG                           203
DW                        20.800 usec
DE                         6.50 usec
TE                        299.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

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NUC1                         13C
P1                          8.50 usec
PL1                        -2.00 dB
PL1W                57.32743073 W
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NOC2                         1H
PCPD2                     80.00 usec
PL2                       -1.00 dB
PL12                      14.26 dB
PL13                      14.46 dB
PL2W                13.18669796 W
PL12W                0.39276794 W
PL13W                0.37509048 W
SFO2                400.1716007 MHz
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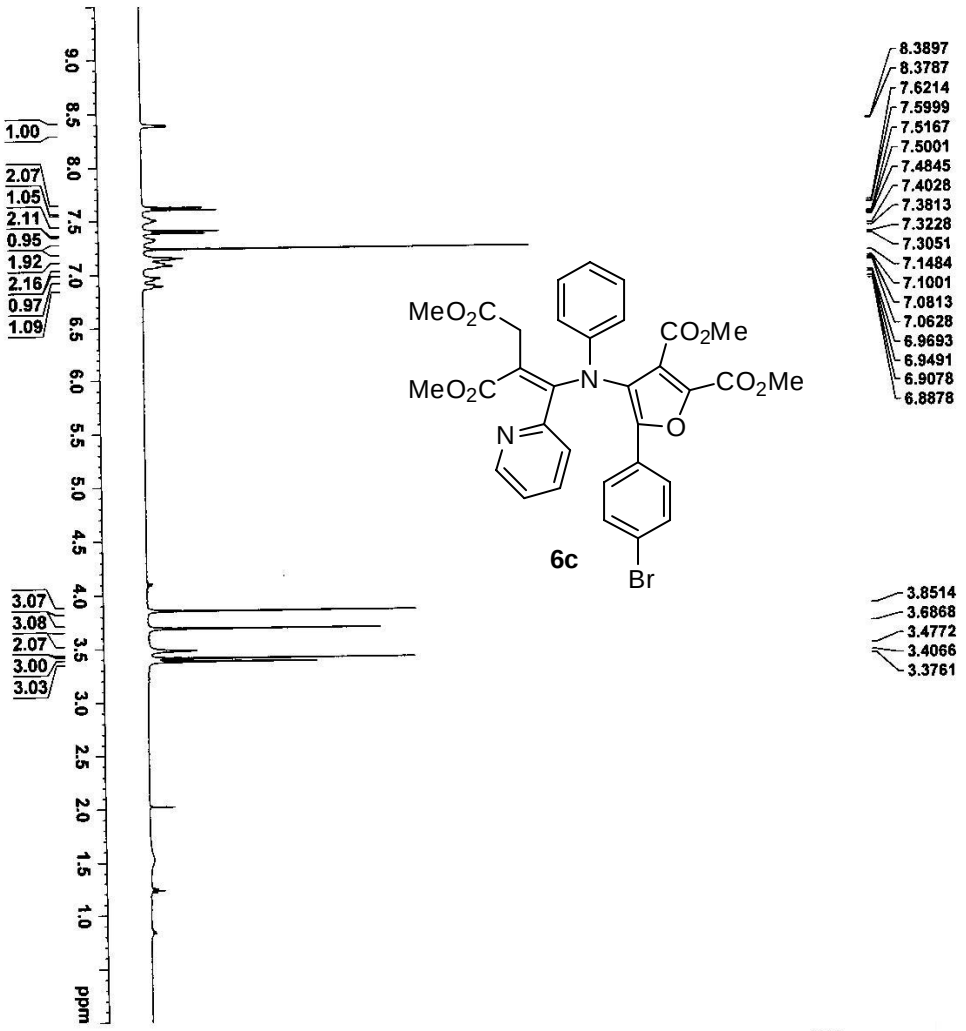


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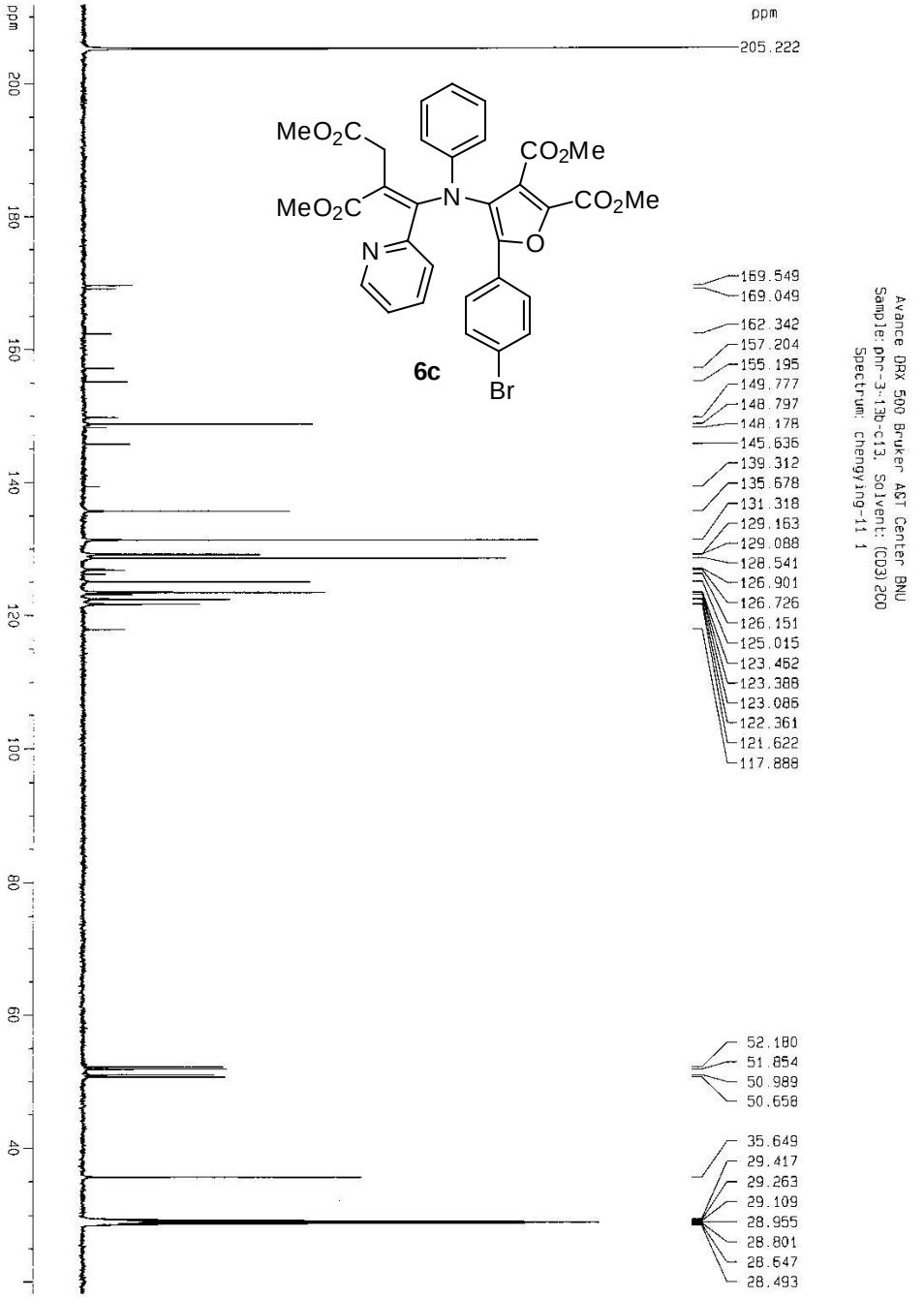
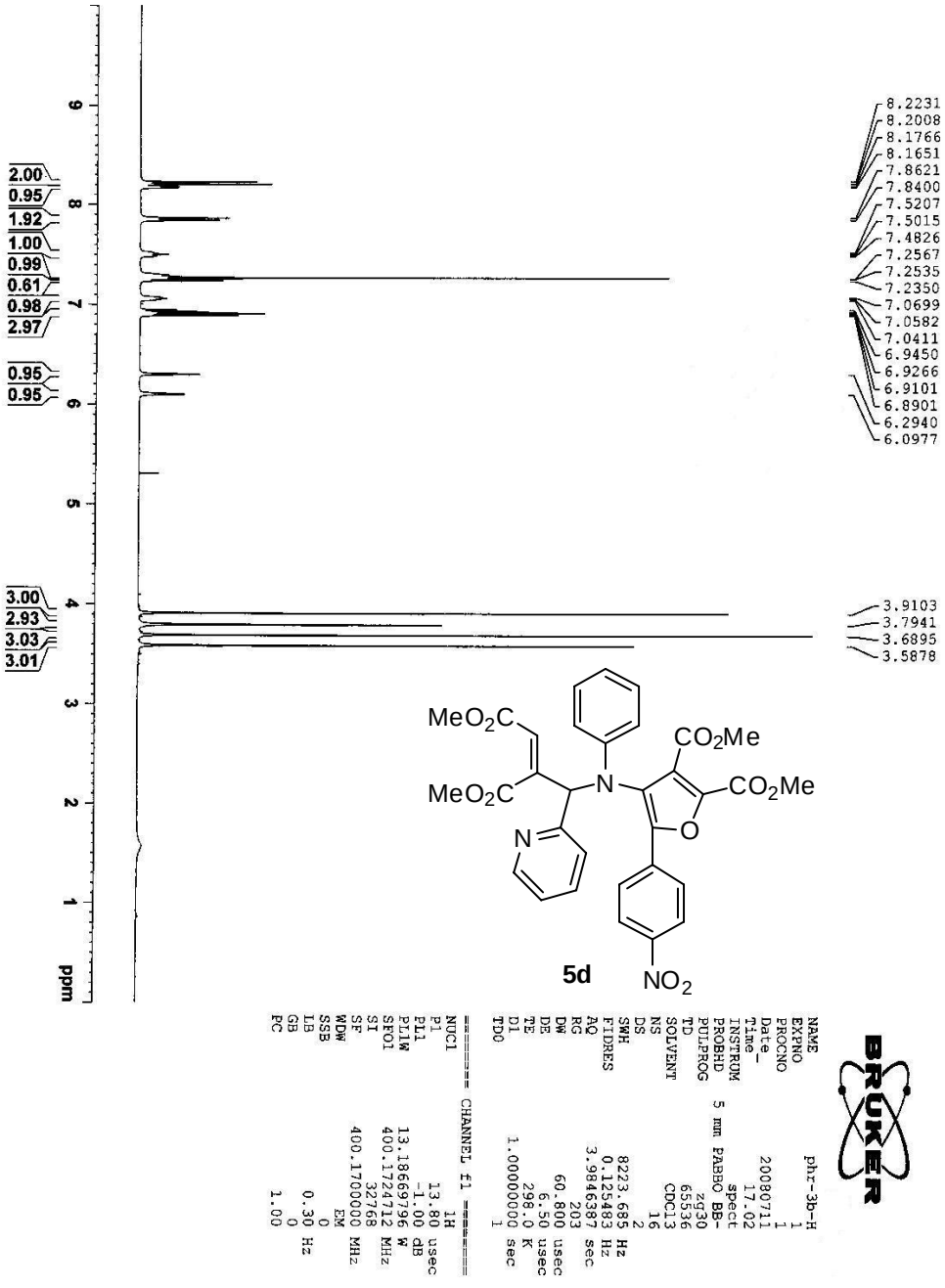
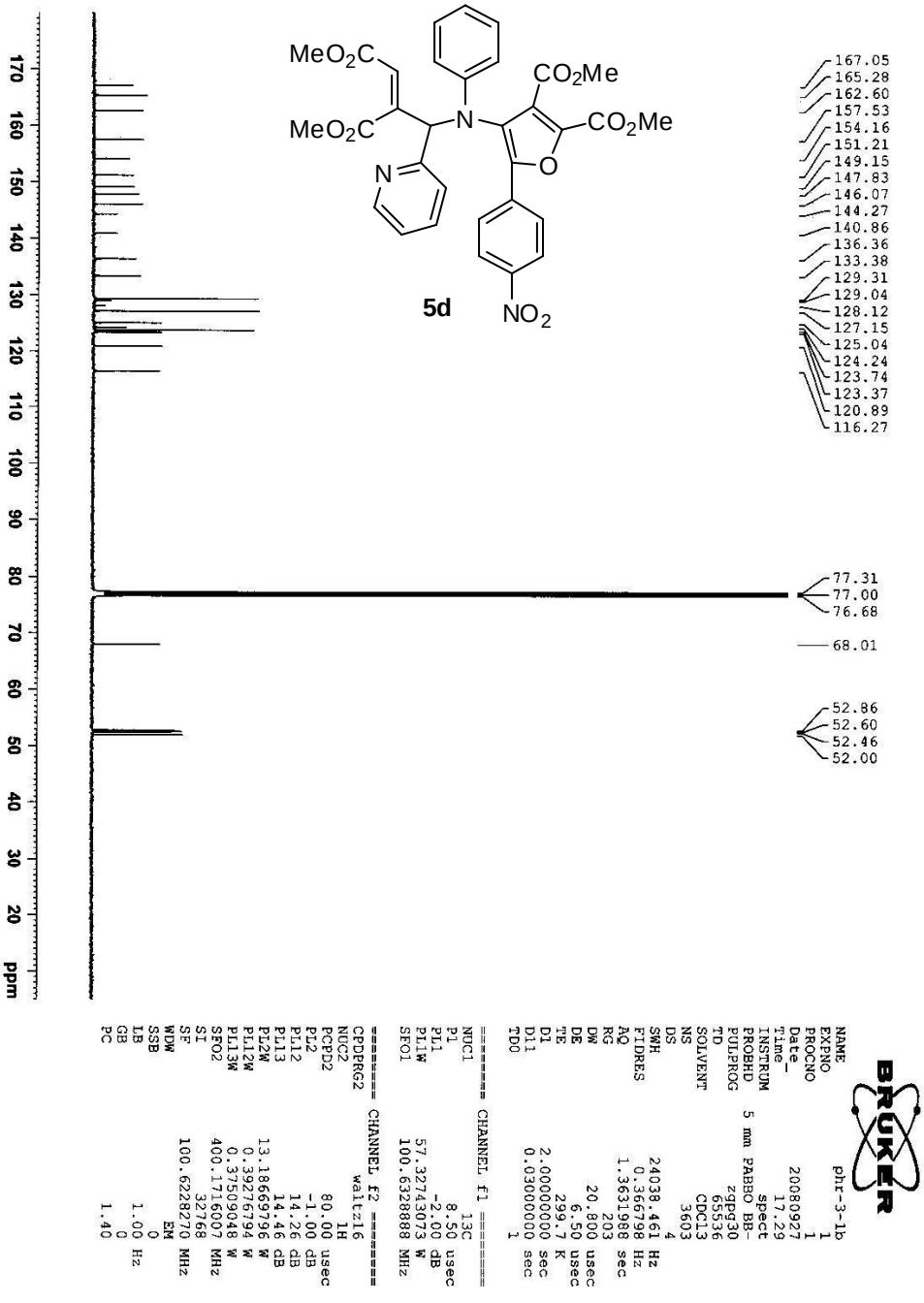
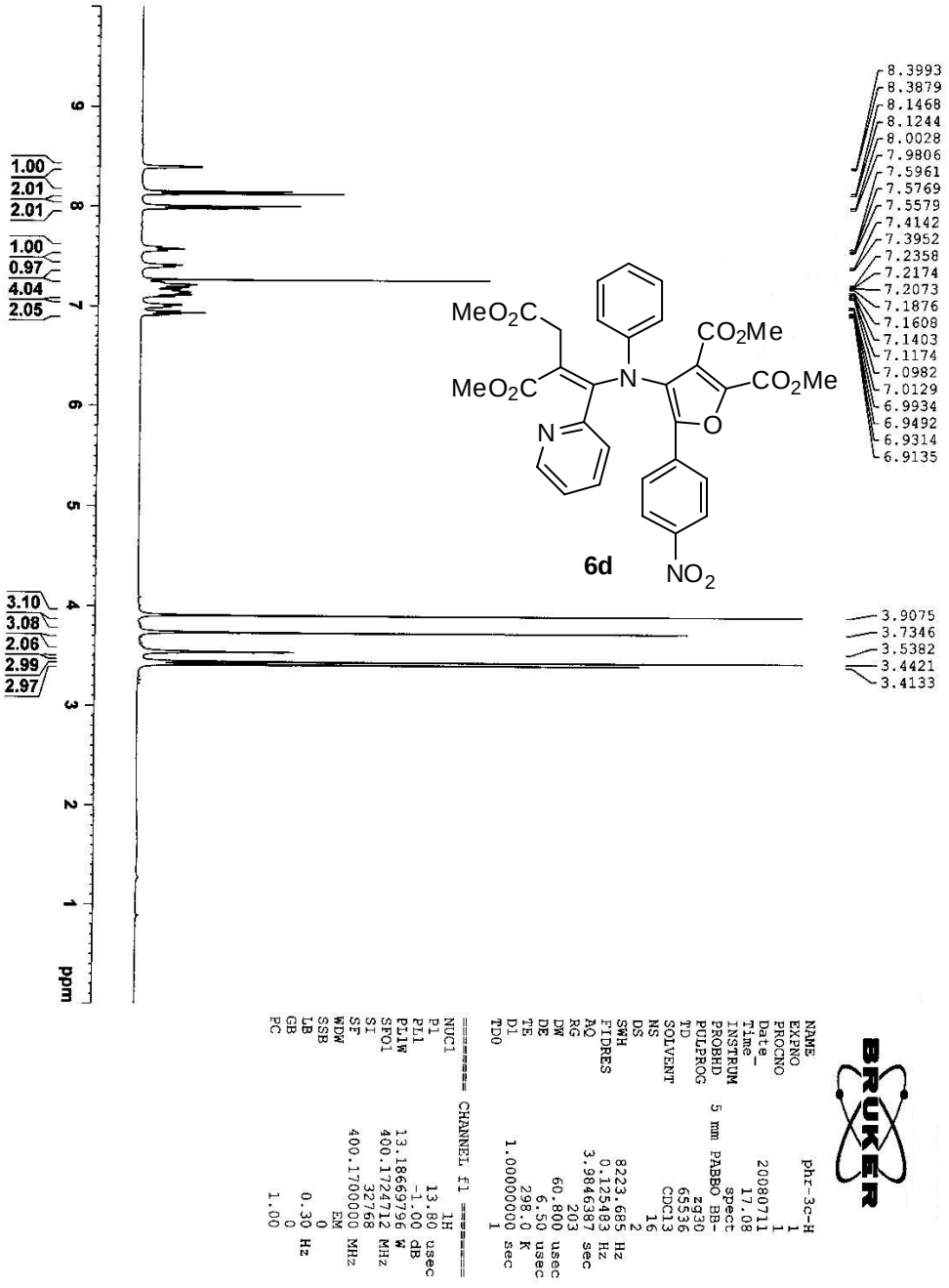
DS                            2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                          203
DW                 60.800 usec
DE                  6.50 usec
TE                  297.6 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       1H
P1                  13.80 usec
PL1                  -1.00 dB
PL1W          13.18669796 W
SFO1          400.1724712 MHz
SI                       32768
SF            400.1700135 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```


ppm
205.222
169.549
169.049
162.342
157.204
155.195
149.777
148.797
148.178
145.636
139.312
135.678
131.318
129.163
129.088
128.541
126.901
126.726
126.151
125.015
123.462
123.388
123.086
122.361
121.622
117.888
52.180
51.854
50.989
50.658
35.649
29.417
29.263
29.109
28.955
28.801
28.647
28.493
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Br
6c
Avance DRX 500 Bruker AST Center BNU
Sample: phr-3-13b-c13, Solvent: (CD3)2CO
Spectrum: chengying-11 1

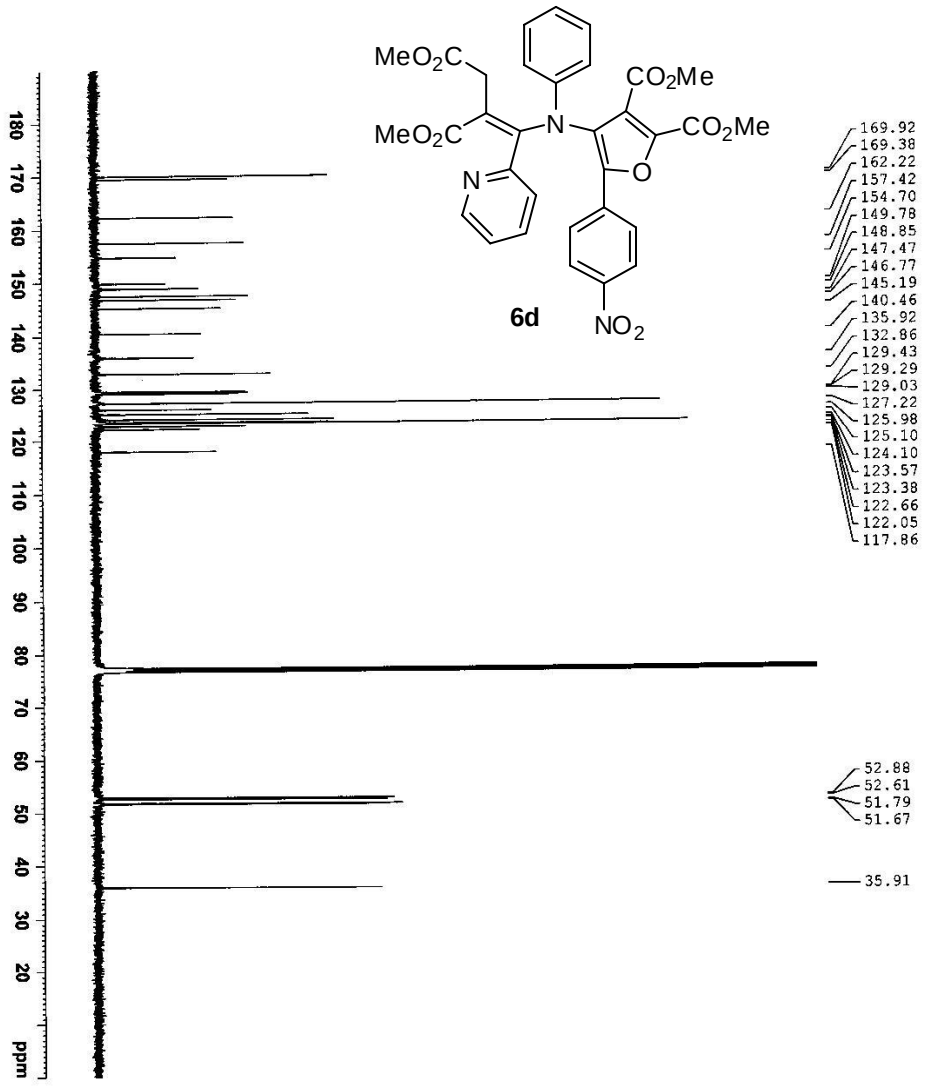

8.2231
8.2008
8.1766
8.1651
7.8621
7.8400
7.5207
7.5015
7.4826
7.2567
7.2535
7.2350
7.0699
7.0582
7.0411
6.9450
6.9266
6.9101
6.8901
6.2940
6.0977
3.9103
3.7941
3.6895
3.5878
2.00
0.95
1.92
1.00
0.99
0.61
0.98
2.97
0.95
0.95
3.00
2.93
3.03
3.01
ppm
MeO2C
MeO2C
N
CO2Me
CO2Me
O
N
NO2
5d
NAME phr-3b-H
EXPNO 1
PROCNO 1
Date_ 20080711
Time 17.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLIW 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
NO2
5d
167.05
165.28
162.60
157.53
154.16
151.21
149.15
147.83
146.07
144.27
140.86
136.36
133.38
129.31
129.04
128.12
127.15
125.04
124.24
123.74
123.37
120.89
116.27
77.31
77.00
76.68
68.01
52.86
52.60
52.46
52.00
ppm
NAME phr-3-1b
EXPNO 1
PROCNO 1
Date_ 20080927
Time_ 17.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3603
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



NAME phr-3c-H
EXPNO 1
PROCNO 1
Date_ 20080711
Time_ 17.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125403 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

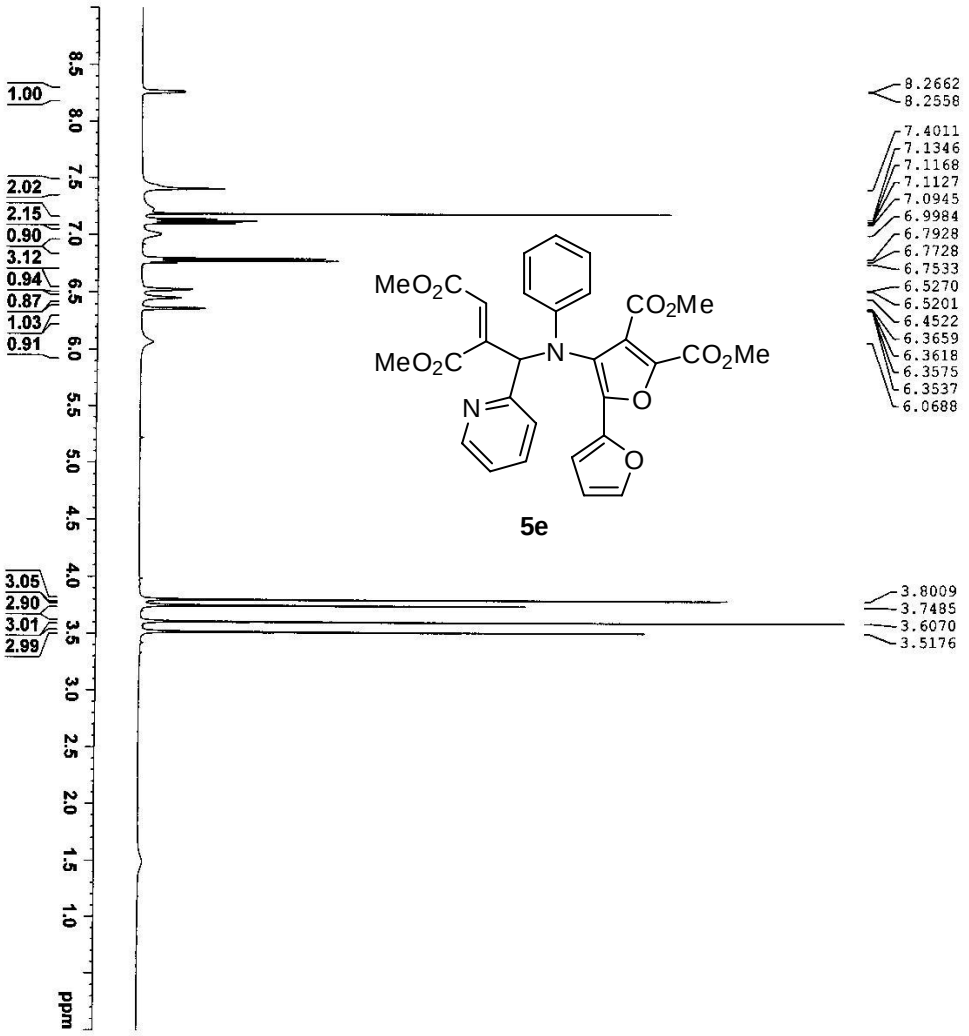
======== CHANNEL f1 ========
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
NAME                                    phr-3c-C13
EXPNO                                            1
PROCNO                                           1
Date_                                     20080711
Time_                                         17.50
INSTRUM                                       spect
PROBHD     5 mm PABBO BB-
PULPROG                                   zgpg30
TD                                            65536
SOLVENT                                     CDCl3
NS                                             3850
DS                                                4
SWH                                       24038.461 Hz
FIDRES                                        0.366798 Hz
AQ                                        1.3631988 sec
RG                                              203
DW                                           20.800 usec
DE                                            6.50 usec
TE                                          299.1 K
D1                                        2.00000000 sec
D11                                       0.03000000 sec
TD0                                               1

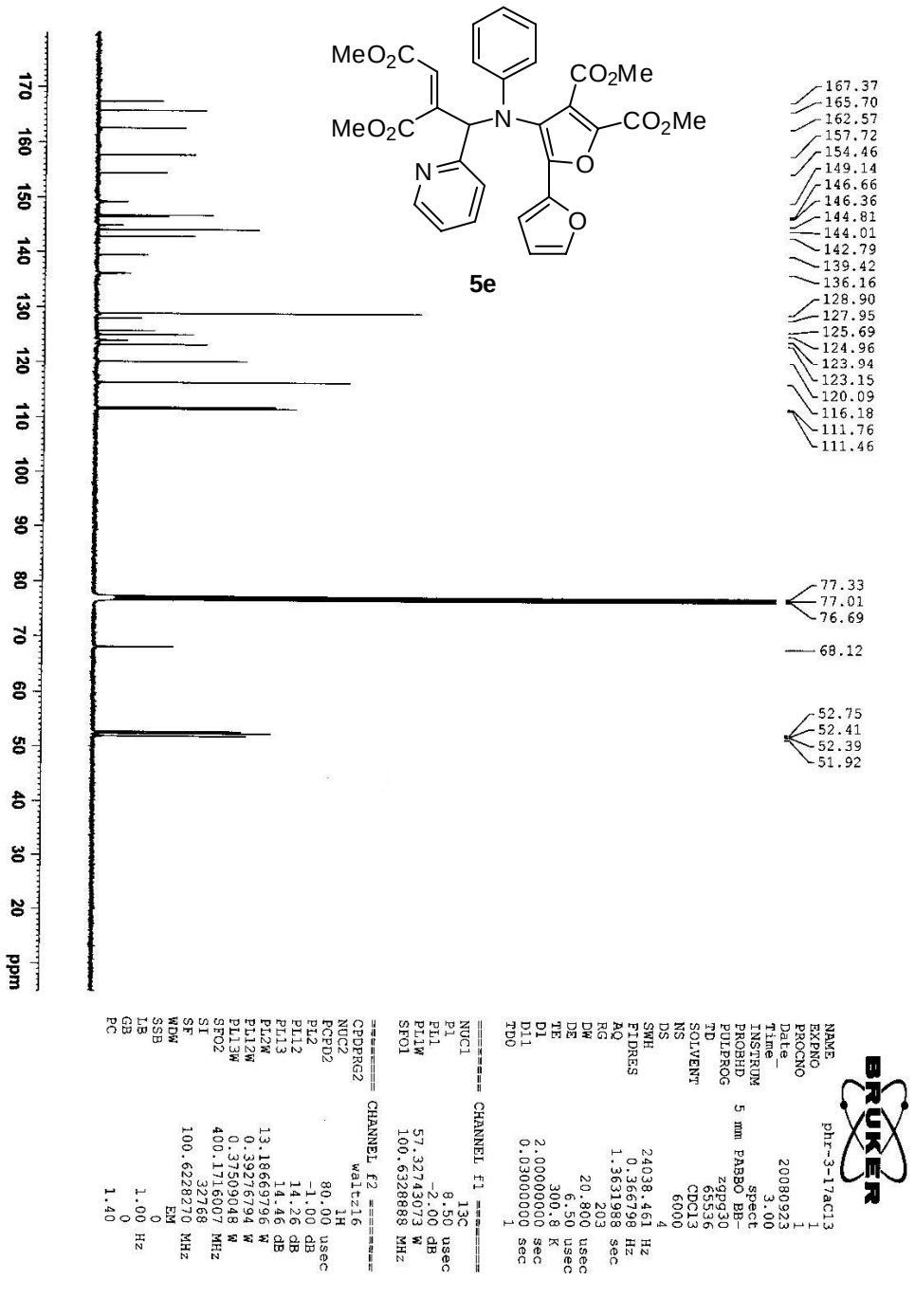
======== CHANNEL f1 ========
NUC1                                            13C
P1                                            8.50 usec
PL1                                          -2.00 dB
PL1W                                      57.32743073 W
SFO1                                      100.6328888 MHz

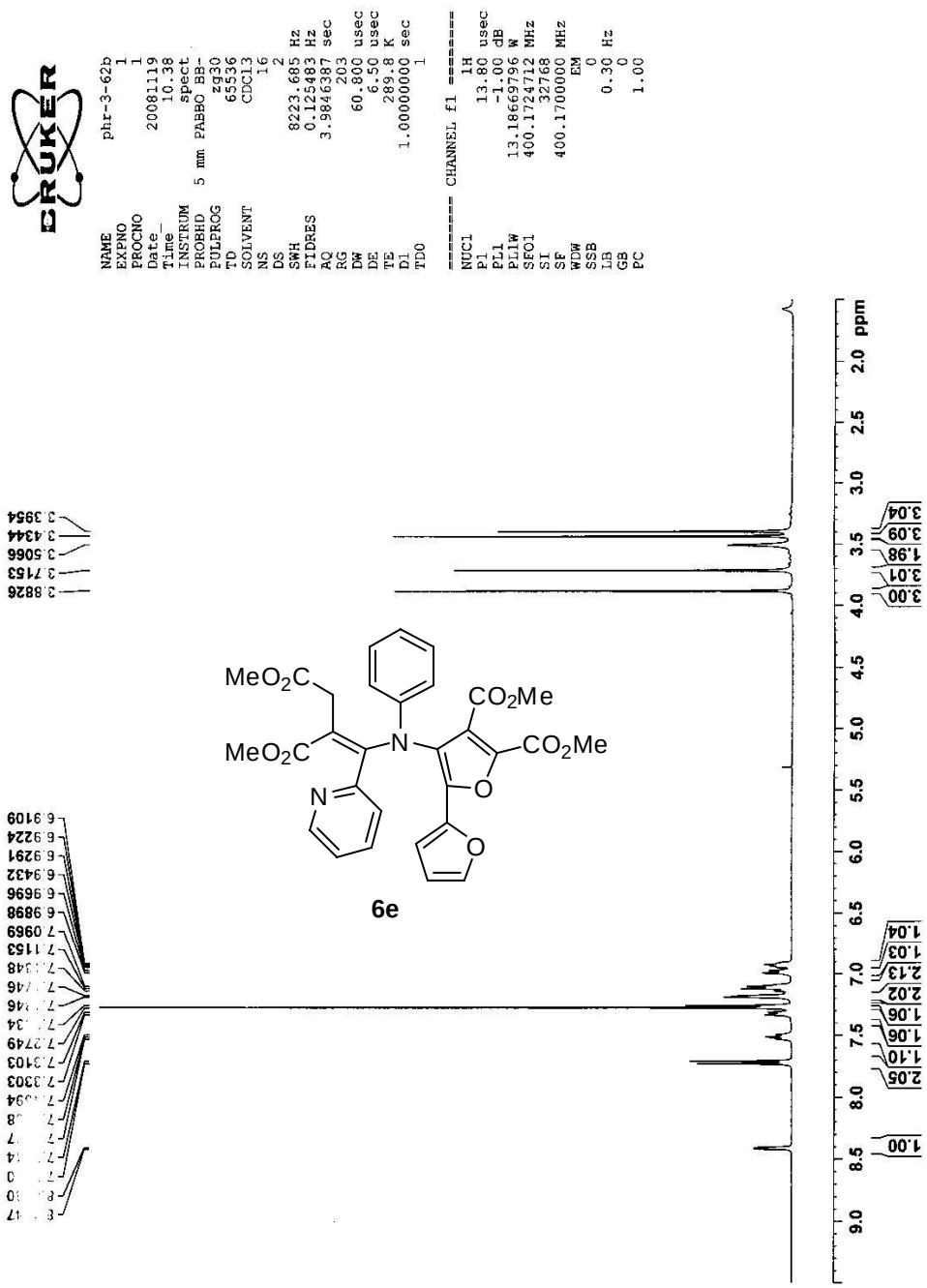
======== CHANNEL f2 ========
CPDPRG2                                     waltz16
NUC2                                            1H
PCPD2                                        80.00 usec
PL2                                          -1.00 dB
PL12                                         14.26 dB
PL13                                         14.46 dB
PL2W                                      13.18669796 W
PL12W                                      0.39276794 W
PL13W                                      0.37509048 W
SFO2                                      400.1716007 MHz
SI                                            32768
SF                                        100.6228270 MHz
WDW                                              EM
SSB                                               0
LB                                             1.00 Hz
GB                                                0
PC                                             1.40
```

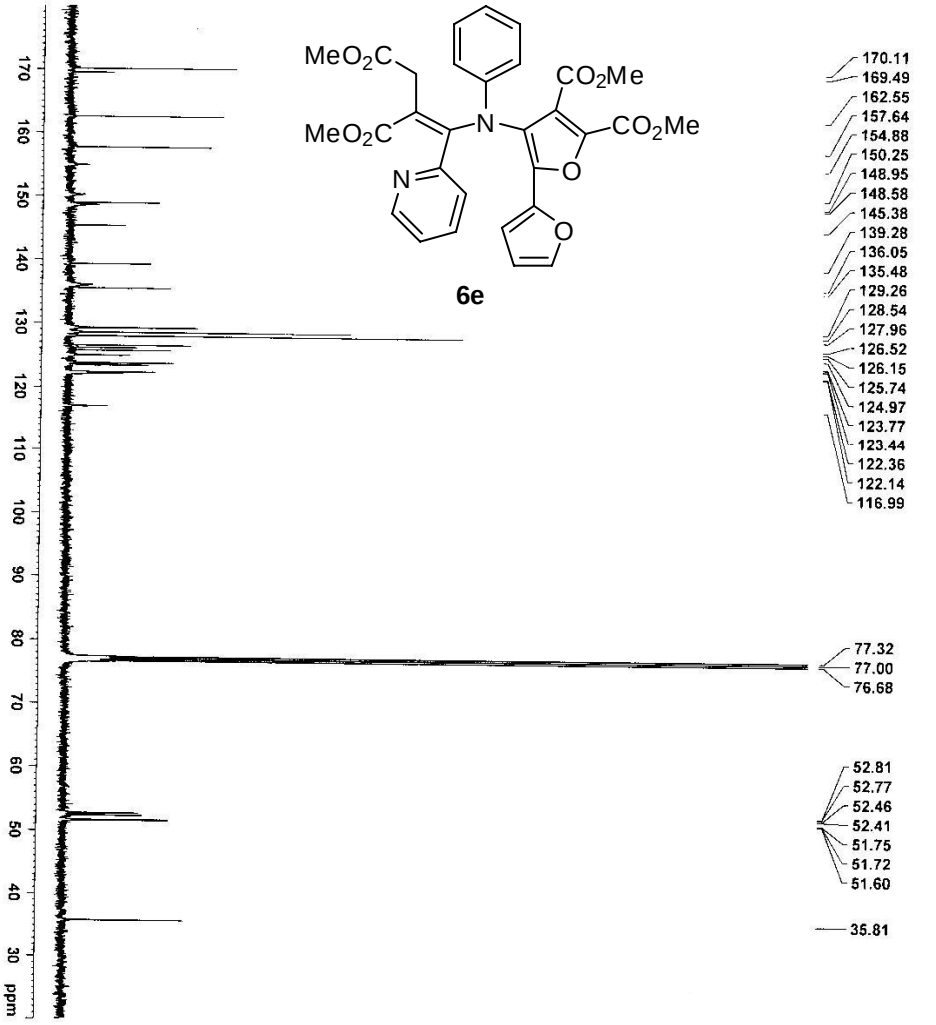


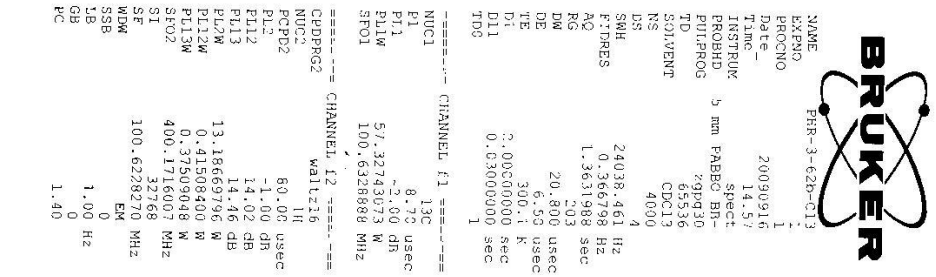
```
NAME                 phr-3-17a
EXPNO                        1
PROCNO                       1
Date_             20080917
Time_                    10.55
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       203
DW                  60.800 usec
DE                   6.50 usec
TE                    298.1 K
D1                 1.00000000 sec
TD0                         1

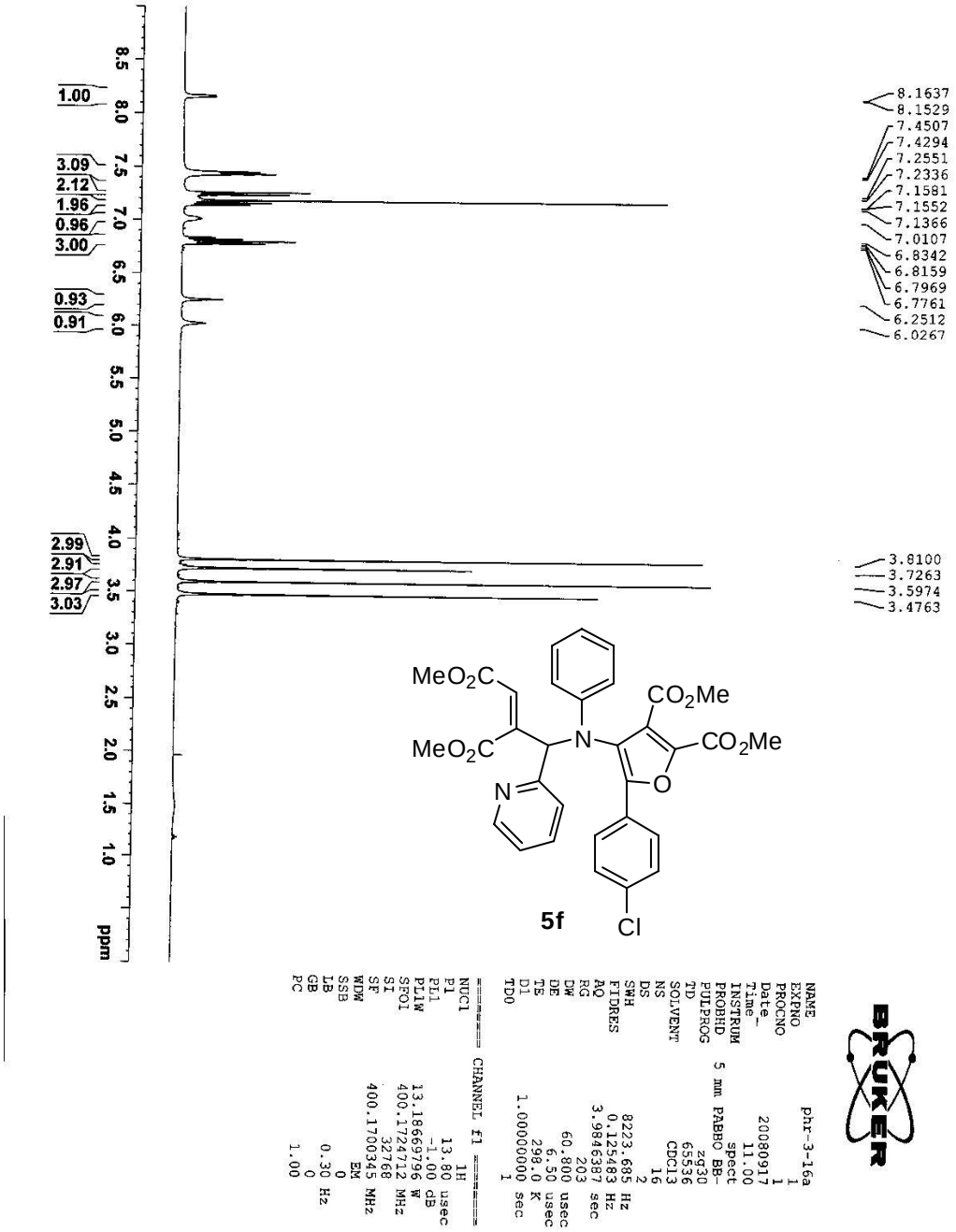
======== CHANNEL f1 ========
NUC1                      1H
P1                     13.80 usec
PL1                    -1.00 dB
PL1W            13.18669796 W
SFO1            400.1724712 MHz
SI                      32768
SF              400.1700350 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

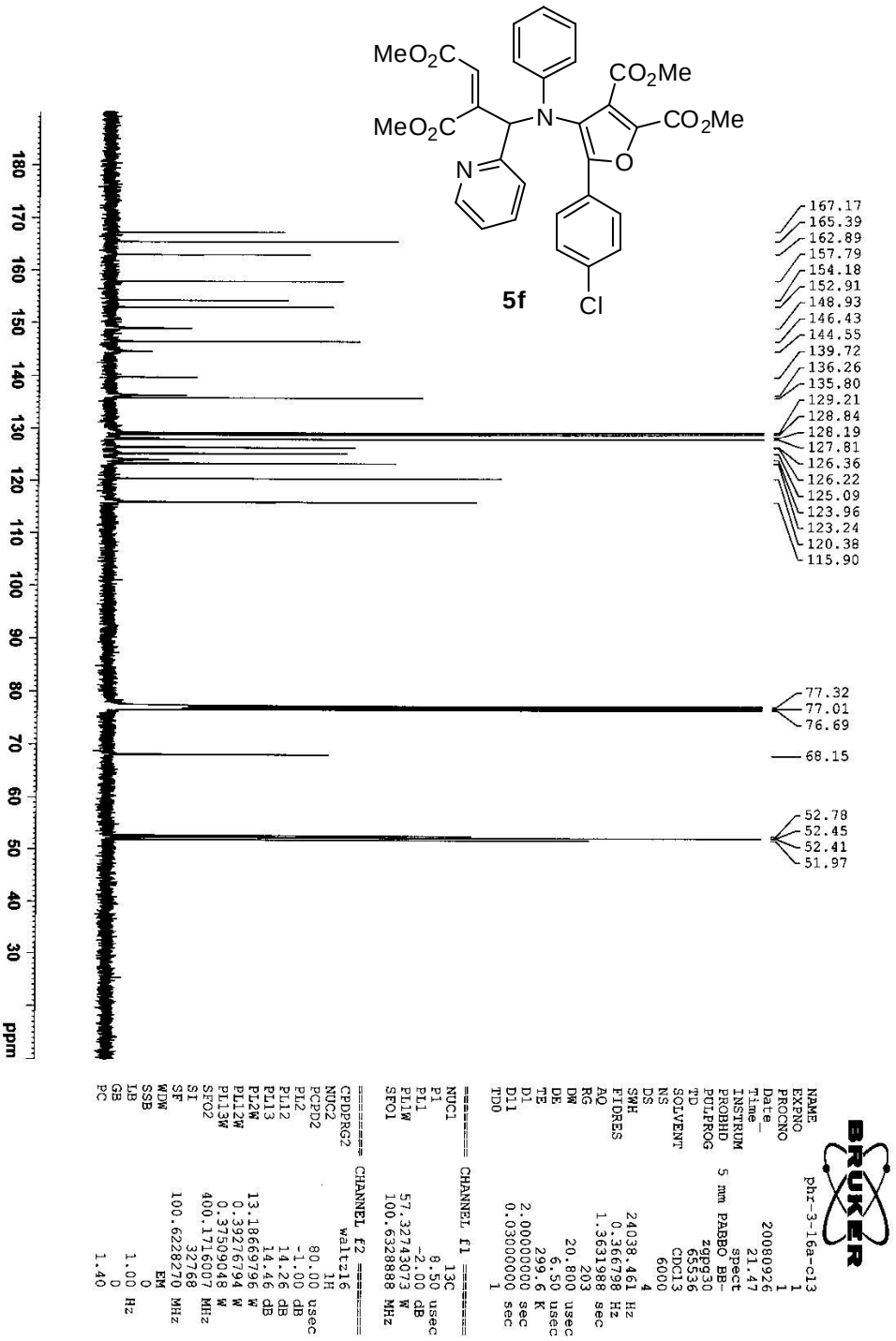

MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
O
5e
167.37
165.70
162.57
157.72
154.46
149.14
146.66
146.36
144.81
144.01
142.79
139.42
136.16
128.90
127.95
125.69
124.96
123.94
123.15
120.09
116.18
111.76
111.46
77.33
77.01
76.69
68.12
52.75
52.41
52.39
51.92
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
NAME phr-3-17ac13
EXPNO 1
PROCNO 1
Date_ 20080923
Time_ 3.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

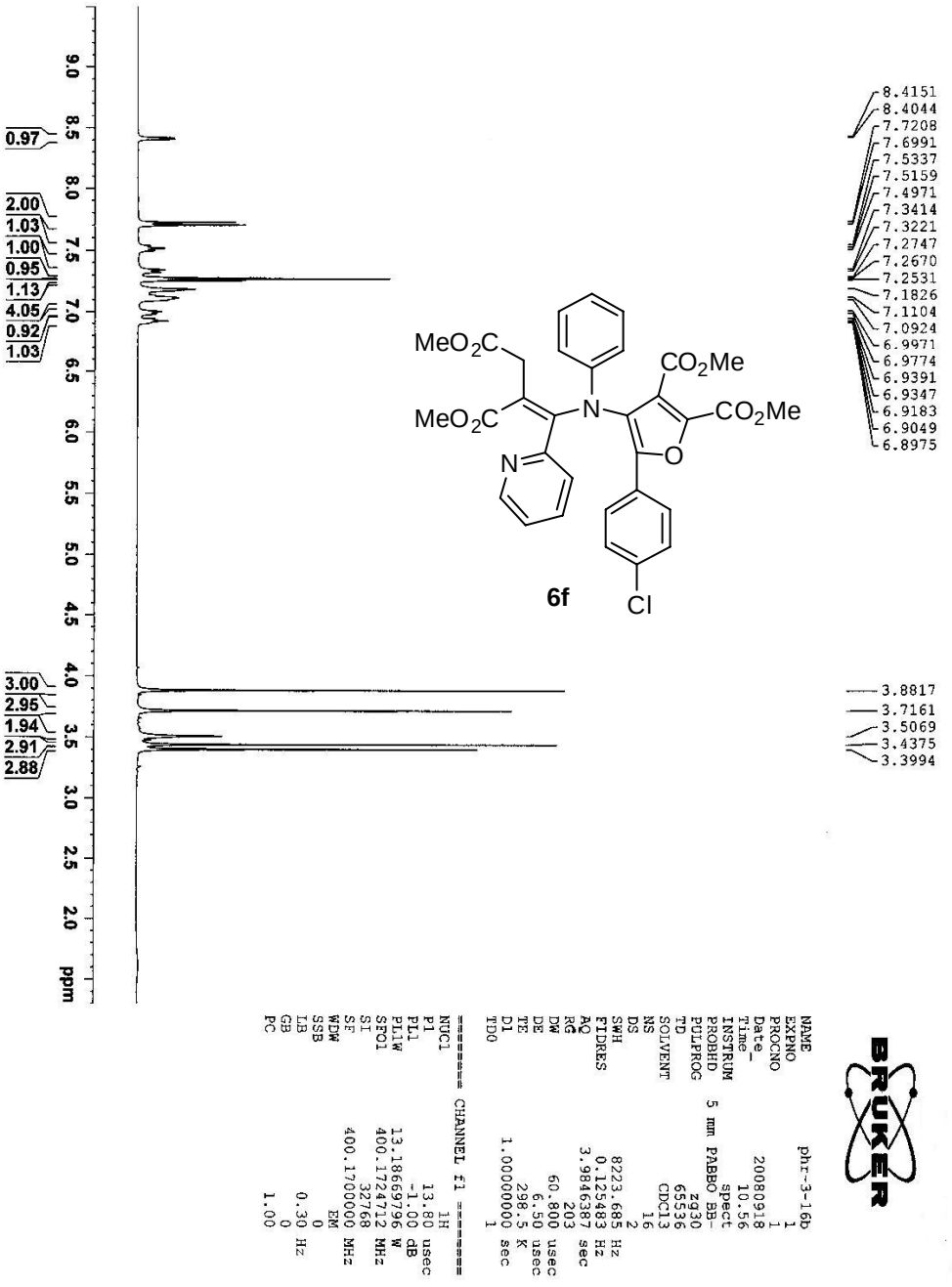

NAME phr-3-62b
EXPNO 1
PROCNO 1
Date_ 20081119
Time 10.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 289.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO2C
CO2Me
MeO2C
CO2Me
N
N
O
O
6e
ppm

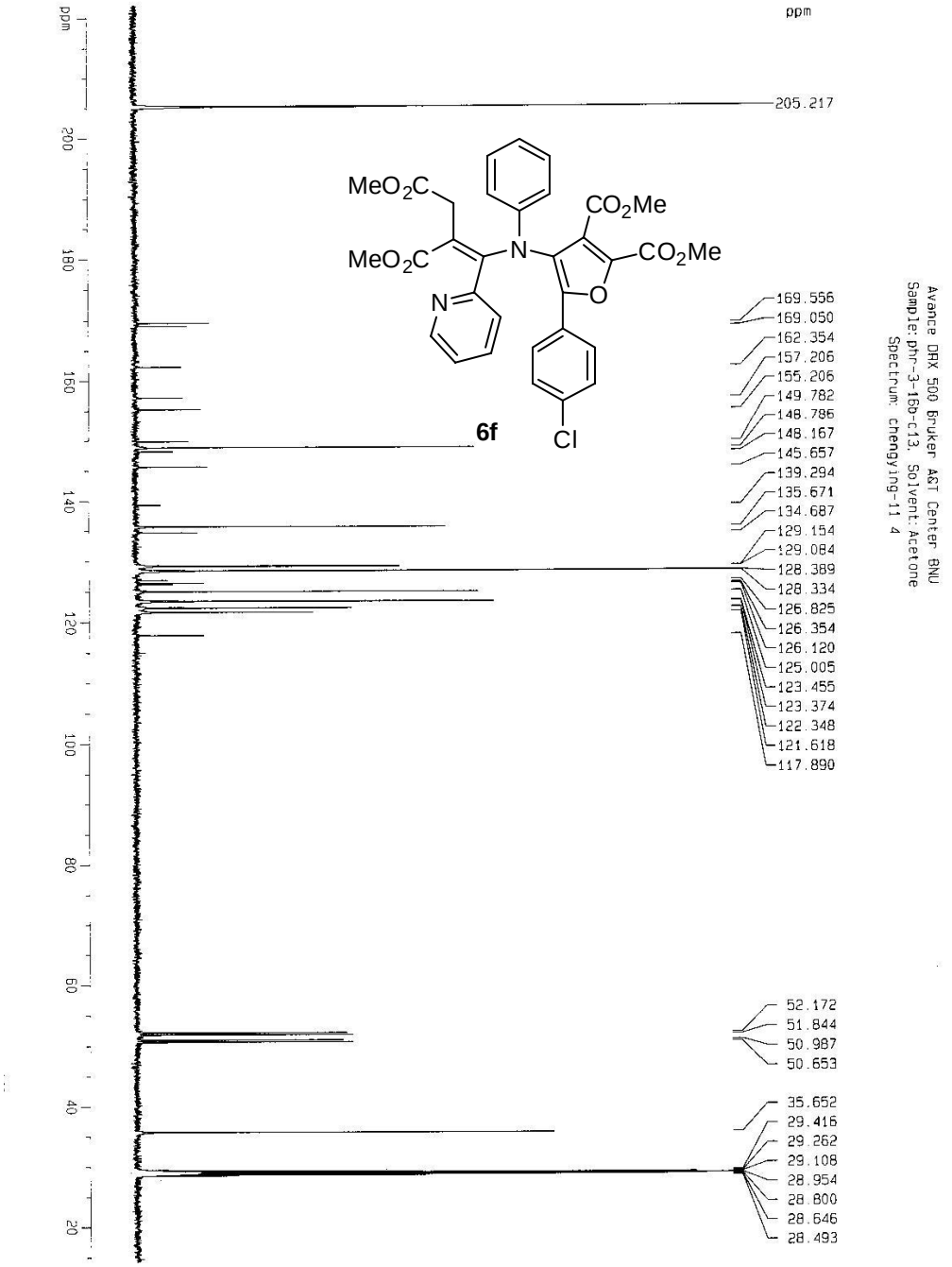

MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
O
6e
170.11
169.49
162.55
157.64
154.88
150.25
148.95
148.58
145.38
139.28
136.05
135.48
129.26
128.54
127.96
126.52
126.15
125.74
124.97
123.77
123.44
122.36
122.14
116.99
77.32
77.00
76.68
52.81
52.77
52.46
52.41
51.75
51.72
51.60
35.81
ppm

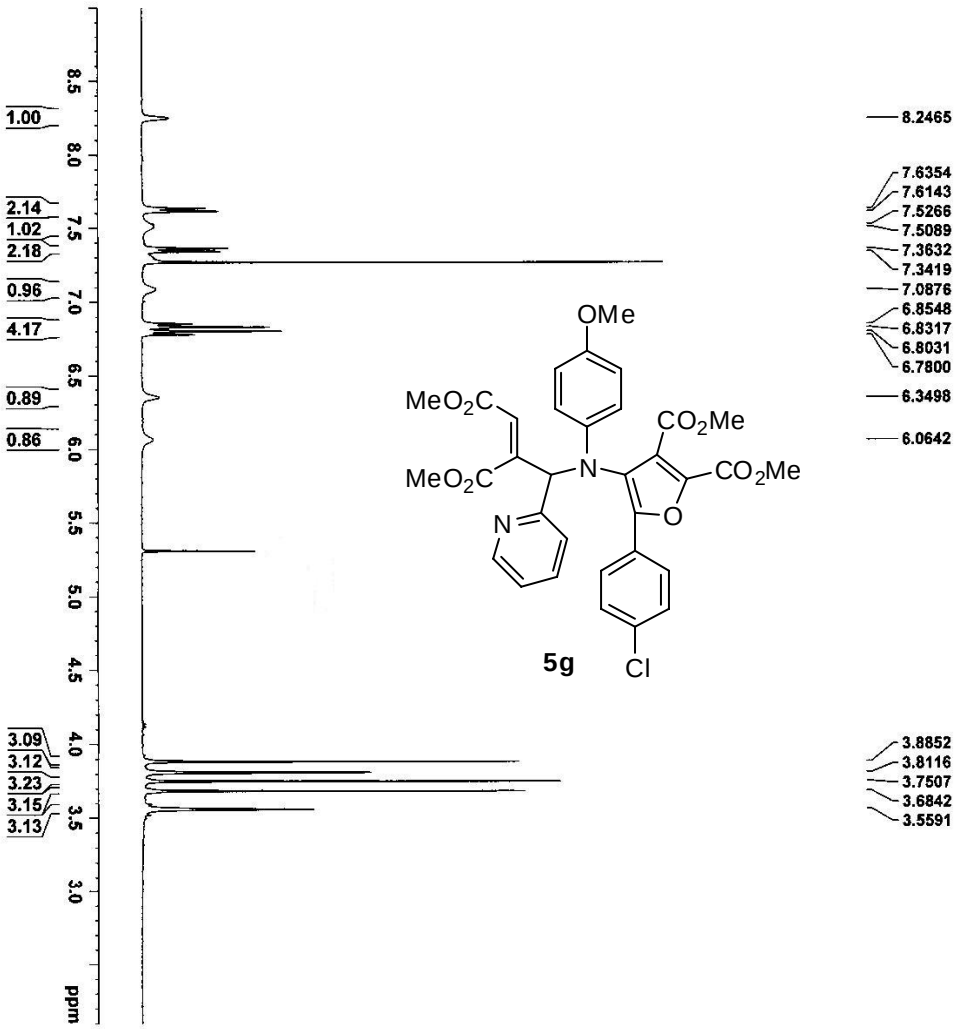

NAME FHR-3-62b-C13
EXPNO 1
PROCNO 1
Date_ 20090916
Time 14.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D0 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6278270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.1637
8.1529
7.4507
7.4294
7.2551
7.2336
7.1581
7.1552
7.1366
7.0107
6.8342
6.8159
6.7969
6.7761
6.2512
6.0267
3.8100
3.7263
3.5974
3.4763
1.00
3.09
2.12
1.96
0.96
3.00
0.93
0.91
2.99
2.91
2.97
3.03
ppm
MeO2C
CO2Me
MeO2C
N
CO2Me
N
O
Cl
5f
NAME phr-3-16a
EXPNO 1
PROCNO 1
Date_ 20080917
Time_ 11.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700345 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
5f
167.17
165.39
162.89
157.79
154.18
152.91
148.93
146.43
144.55
139.72
136.26
135.80
129.21
128.84
128.19
127.81
126.36
126.22
125.09
123.96
123.24
120.38
115.90
77.32
77.01
76.69
68.15
52.78
52.45
52.41
51.97
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30
ppm
NAME phr-3-16a-c13
EXPNO 1
PROCNO 1
Date_ 20080926
Time_ 21.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

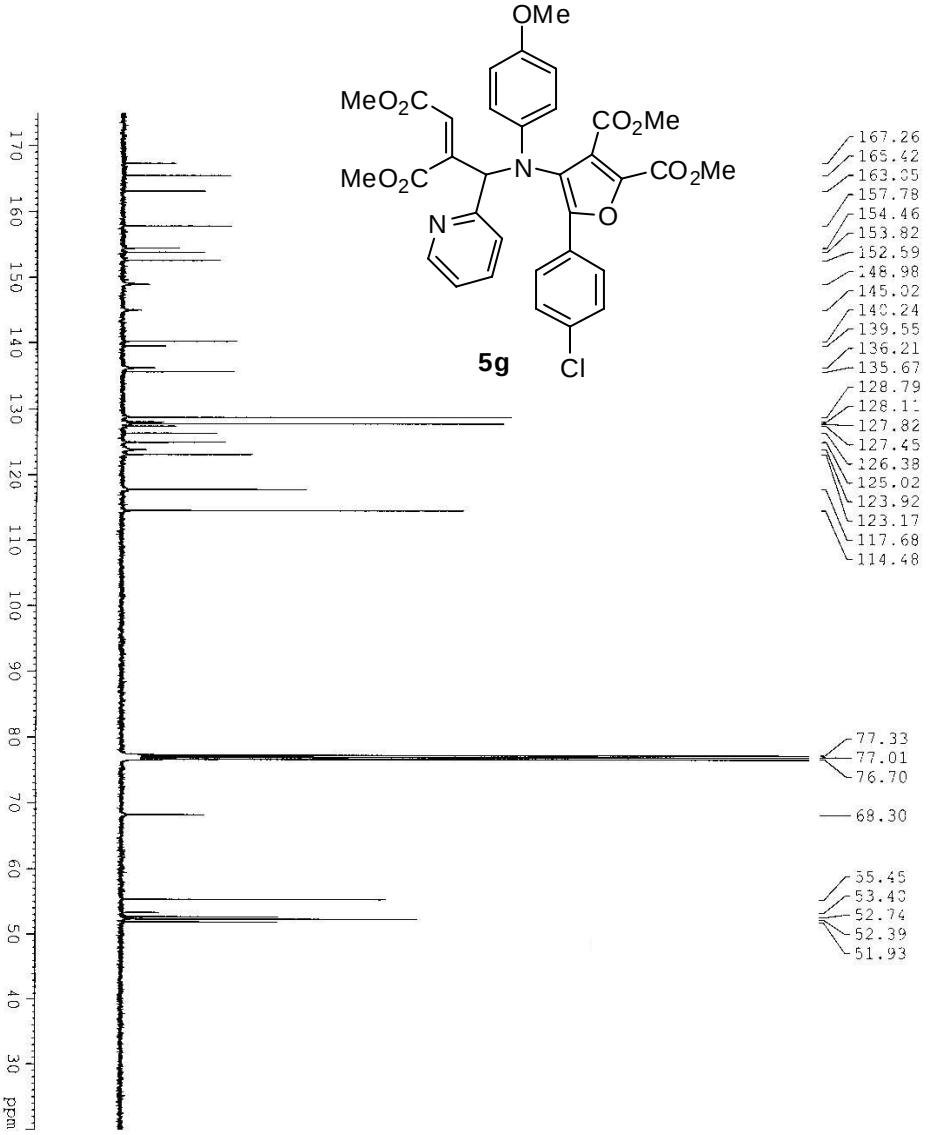

8.4151
8.4044
7.7208
7.6991
7.5337
7.5159
7.4971
7.3414
7.3221
7.2747
7.2670
7.2531
7.1826
7.1104
7.0924
6.9971
6.9774
6.9391
6.9347
6.9183
6.9049
6.8975
3.8817
3.7161
3.5069
3.4375
3.3994
0.97
2.00
1.03
1.00
0.95
1.13
4.05
0.92
1.03
3.00
2.95
1.94
2.91
2.88
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
MeO2C
MeO2C
N
CO2Me
CO2Me
O
N
Cl
6f
NAME phr-3-16b
EXPNO 1
PROCNO 1
Date_ 20080918
Time_ 10.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ppm
205.217
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
6f
169.556
169.050
162.354
157.206
155.206
149.782
148.786
148.167
145.657
139.294
135.671
134.687
129.154
129.084
128.389
128.334
126.825
126.354
126.120
125.005
123.455
123.374
122.348
121.618
117.890
52.172
51.844
50.987
50.653
35.652
29.416
29.262
29.108
28.954
28.800
28.646
28.493
Avance DRX 500 Bruker A&T Center BNU
Sample:phr-3-16b-c13, Solvent:Acetone
Spectrum: chengying-11 4




```
NAME                 phr-3-34a
EXPNO                        1
PROCNO                       1
Date_              20081107
Time                    16.59
INSTRUM                 spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       203
DW                    60.800 usec
DE                     6.50 usec
TE                     295.0 K
D1                  1.00000000 sec
TD0                          1

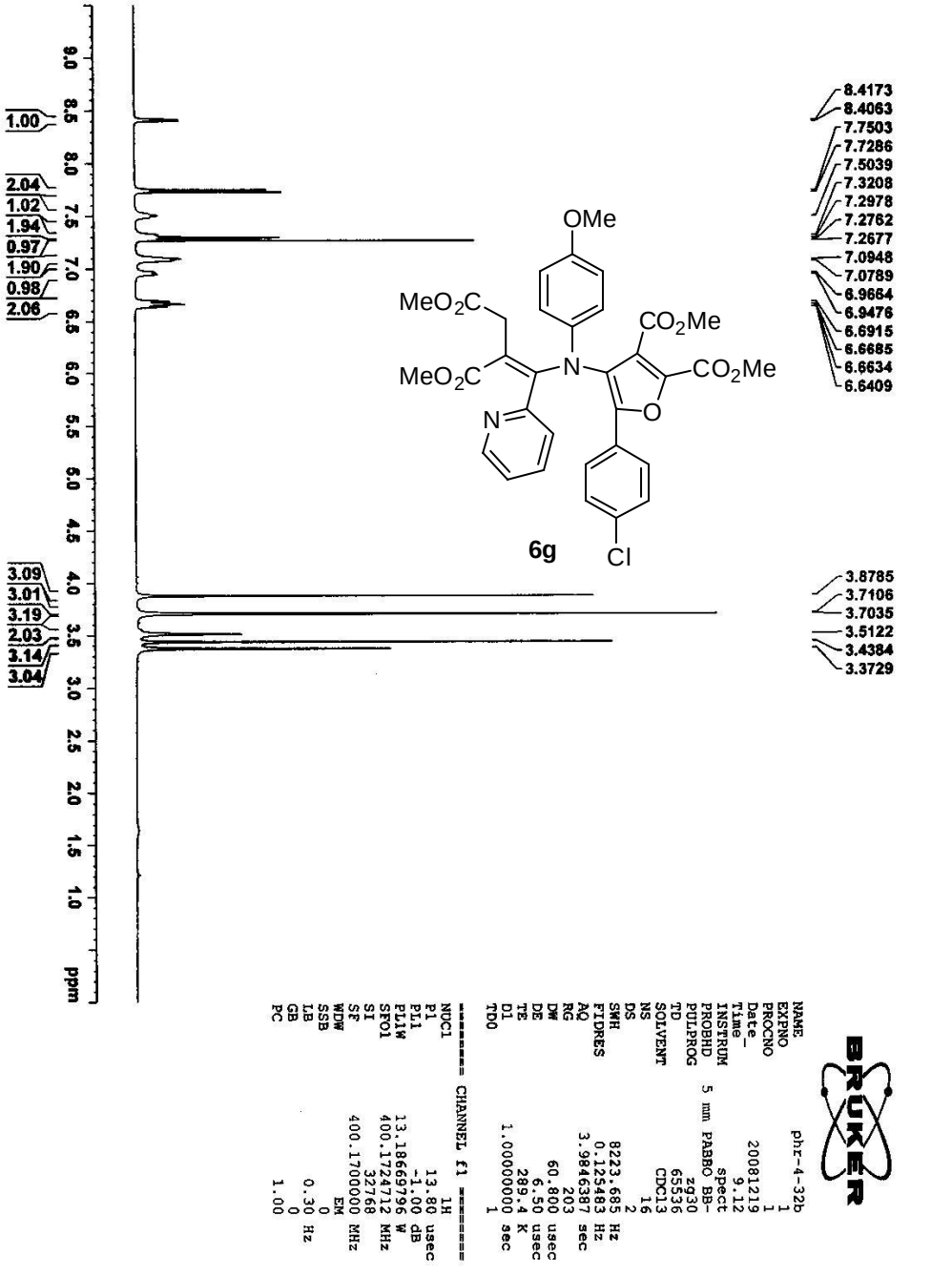
======== CHANNEL f1 ========
NUC1                       1H
P1                      13.80 usec
PL1                     -1.00 dB
PLIW                13.18669796 W
SFO1               400.1724712 MHz
SI                       32768
SF                 400.1700000 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```




```
NAME                 phr-3-34a-C13
EXPNO                           1
PROCNO                          1
Date_                    20081219
Time_                       14.06
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2685
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                          203
DW                       20.800 usec
DE                        6.50 usec
TE                       297.5 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                         13C
P1                          8.50 usec
PL1                        -2.00 dB
PL1W                57.32743073 W
SFO1                100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                            1H
PCPD2                      80.00 usec
PL2                        -1.00 dB
PL12                       14.26 dB
PL13                       14.46 dB
PL2W                13.18669796 W
PL12W                0.39276794 W
PL13W                0.37509048 W
SFO2                400.1716007 MHz
SI                         32768
SF                  100.6228270 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```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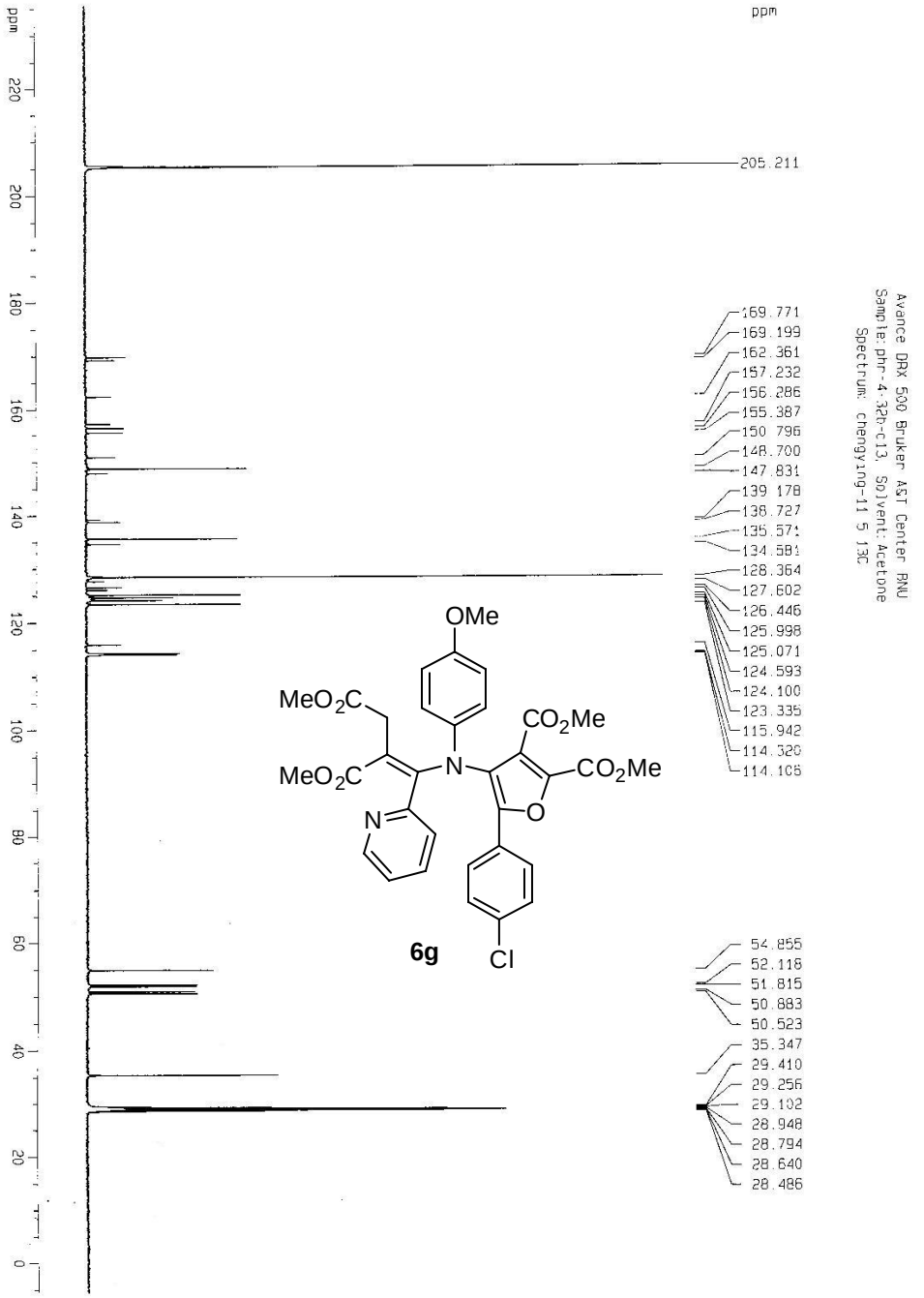
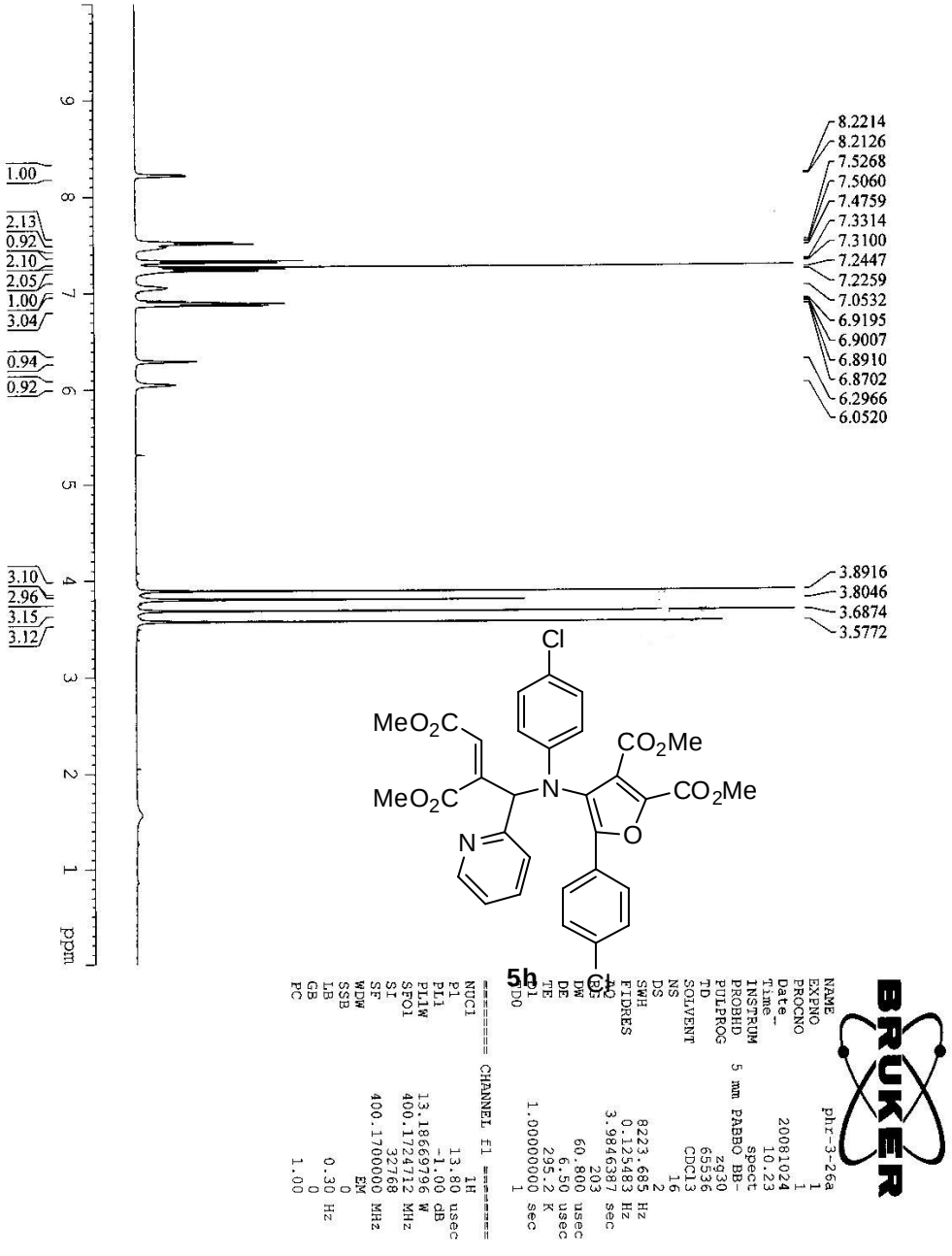
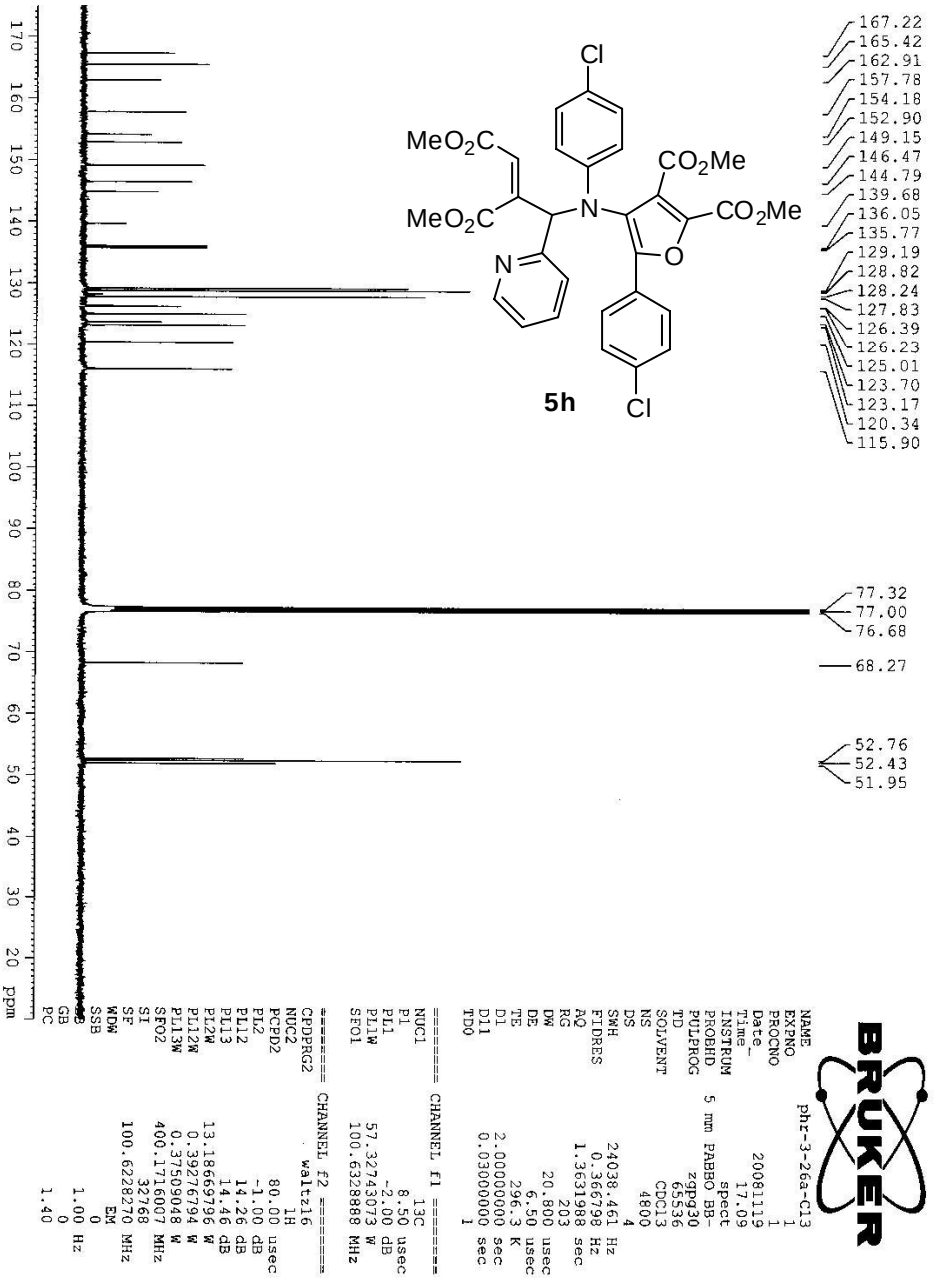
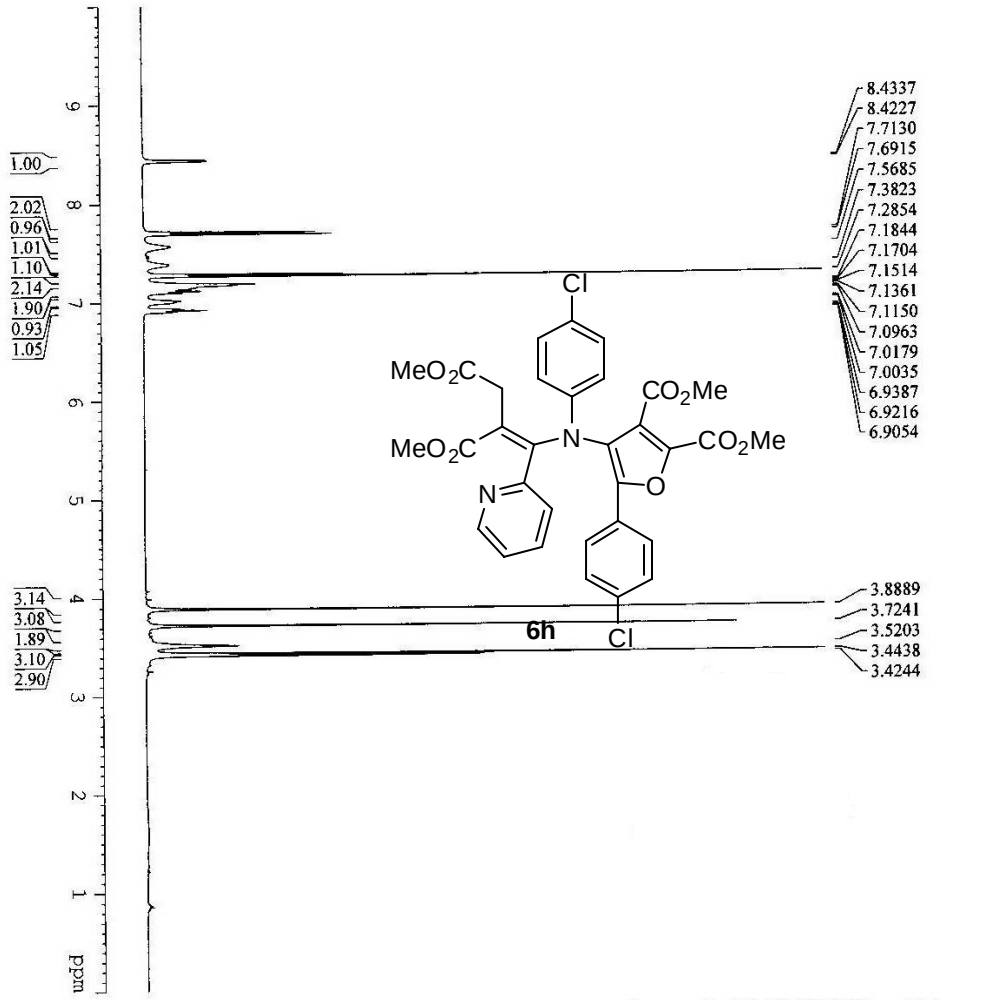
8.4063
7.7503
7.7286
7.5039
7.3208
7.2978
7.2762
7.2677
7.0948
7.0789
6.9664
6.9476
6.6915
6.6685
6.6634
6.6409
3.8785
3.7106
3.7035
3.5122
3.4384
3.3729
1.00
2.04
1.02
1.94
0.97
1.90
0.98
2.06
3.09
3.01
3.19
2.03
3.14
3.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
OMe
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
6g
NAME phr-4-32b
EXPNO 1
PROCNO 1
Date_ 20081219
Time_ 9.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 289.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLW1 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


ppm
205.211
169.771
169.199
162.361
157.232
156.286
155.387
150.796
148.700
147.831
139.178
138.727
135.571
134.581
128.364
127.602
126.446
125.998
125.071
124.593
124.100
123.335
115.942
114.320
114.105
54.855
52.118
51.815
50.883
50.523
35.347
29.410
29.256
29.102
28.948
28.794
28.640
28.486
Avance DRX 500 Bruker AST Center RNU
Sample:phr-4-32h-c13, Solvent: Acetone
Spectrum: changying-11 5 13C
OMe
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
6g


8.2214
8.2126
7.5268
7.5060
7.4759
7.3314
7.3100
7.2447
7.2259
7.0532
6.9195
6.9007
6.8910
6.8702
6.2966
6.0520
3.8916
3.8046
3.6874
3.5772
1.00
2.13
0.92
2.10
2.05
1.00
3.04
0.94
0.92
3.10
2.96
3.15
3.12
ppm
Cl
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
5b
NAME phr-3-26a
EXPNO 1
PROCNO 1
Date_ 20081024
Time_ 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.0000000 sec
D0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLIW 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


5h
Cl
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
167.22
165.42
162.91
157.78
154.18
152.90
149.15
146.47
144.79
139.68
136.05
135.77
129.19
128.82
128.24
127.83
126.39
126.23
125.01
123.70
123.17
120.34
115.90
77.32
77.00
76.68
68.27
52.76
52.43
51.95
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
BRUKER
phr-3-26a-C13
NAME
EXPNO 1
PROCNO 1
Date_ 20081119
Time 17.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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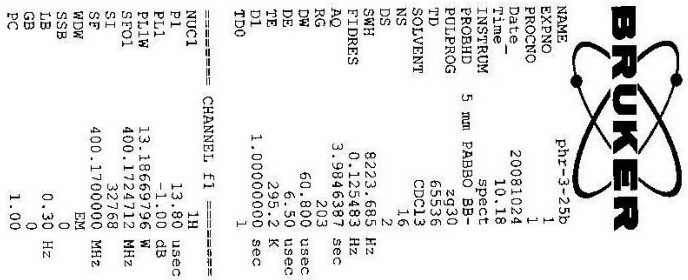
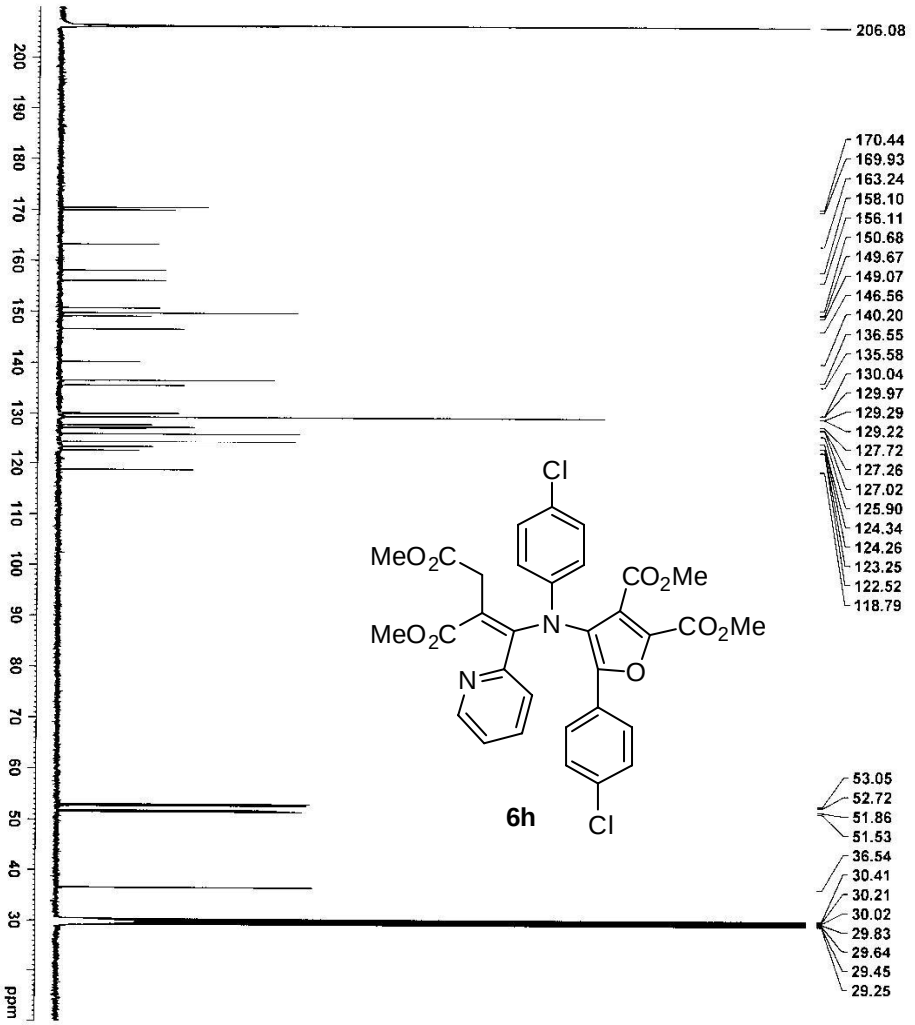
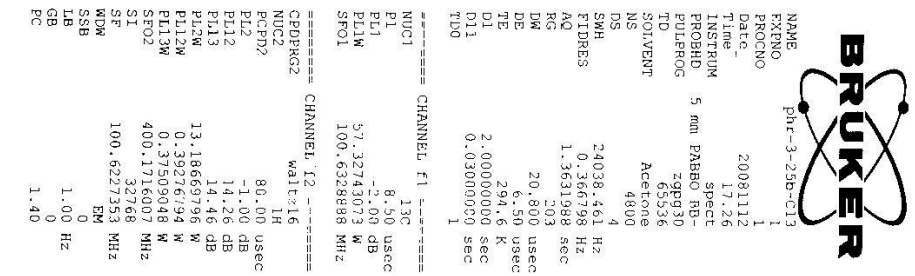
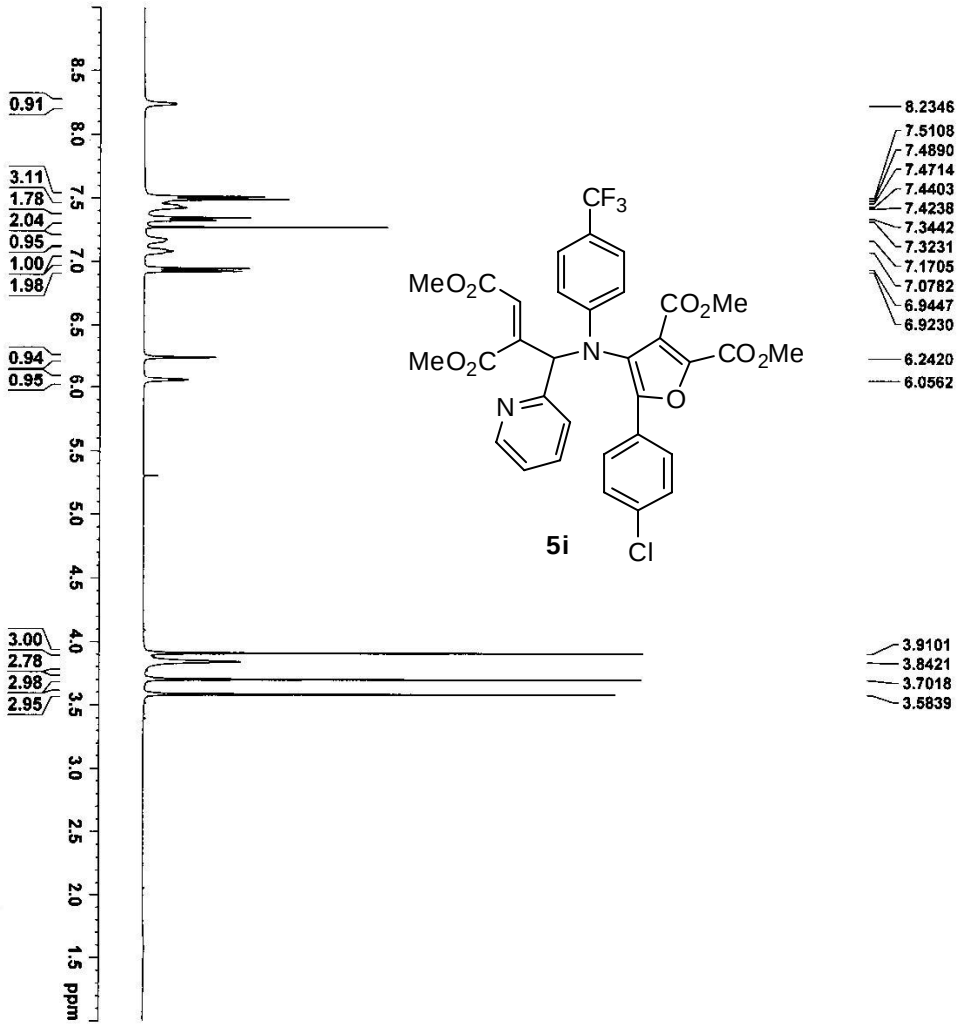
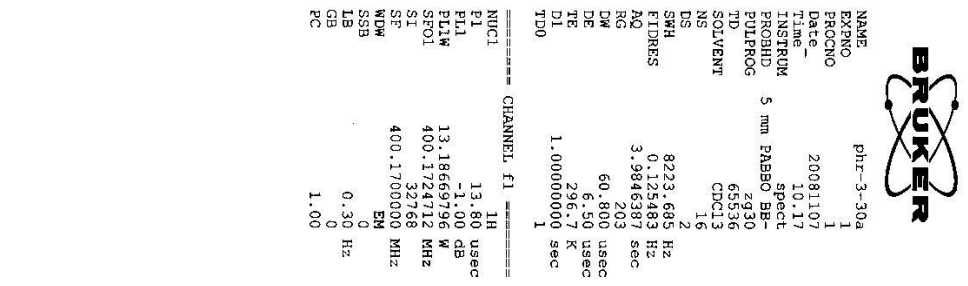
8.4227
7.7130
7.6915
7.5685
7.3823
7.2854
7.1844
7.1704
7.1514
7.1361
7.1150
7.0963
7.0179
7.0035
6.9387
6.9216
6.9054
3.8889
3.7241
3.5203
3.4438
3.4244
1.00
2.02
0.96
1.01
1.10
2.14
1.90
0.93
1.05
3.14
3.08
1.89
3.10
2.90
ppm
Cl
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
6h


BRUKER
NAME phr-3-25b
EXPNO 1
PROCNO 1
Date_ 20081024
Time_ 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


206.08
170.44
169.93
163.24
158.10
156.11
150.68
149.67
149.07
146.56
140.20
136.55
135.58
130.04
129.97
129.29
129.22
127.72
127.26
127.02
125.90
124.34
124.26
123.25
122.52
118.79
53.05
52.72
51.86
51.53
36.54
30.41
30.21
30.02
29.83
29.64
29.45
29.25
ppm
Cl
MeO2C
CO2Me
MeO2C
N
CO2Me
N
O
6h
Cl

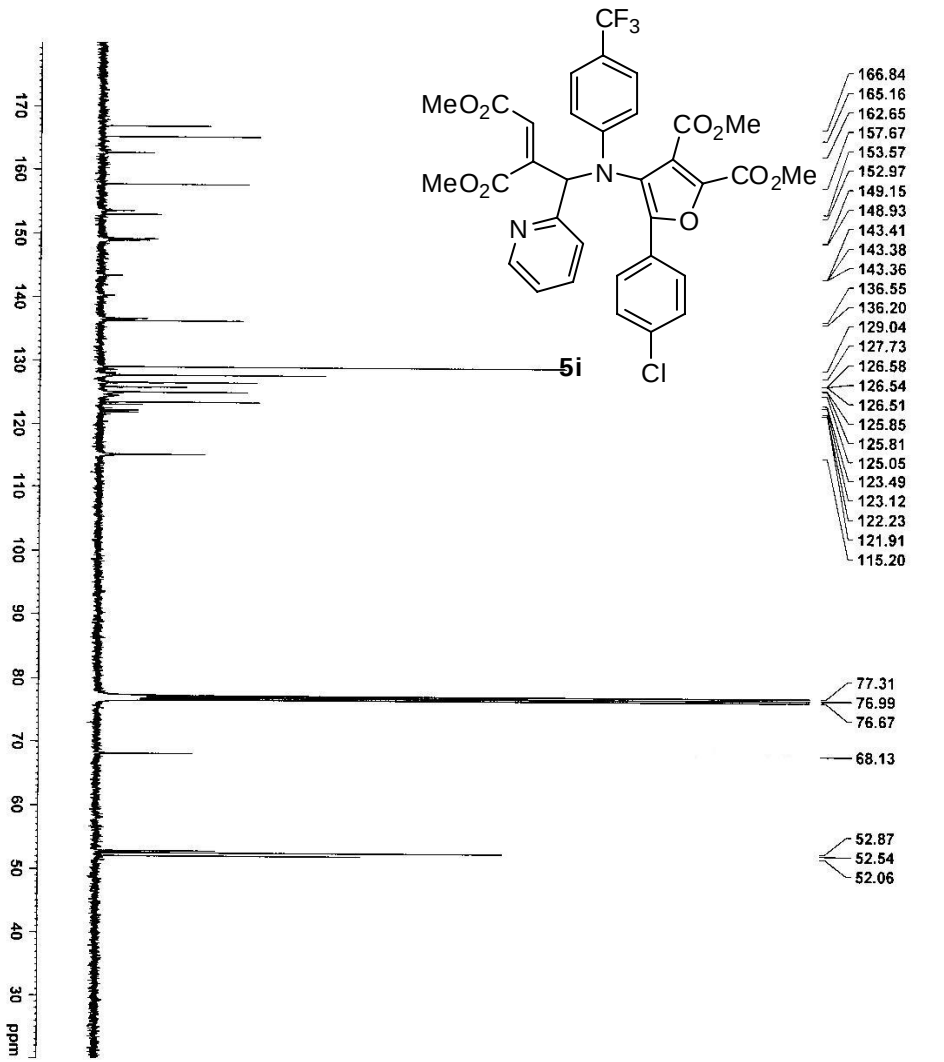

NAME phr-3-25b-C13
FXPNO 1
PROCNO 1
Date_ 20081112
Time_ 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 4800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6227353 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

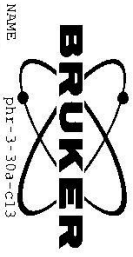




```
NAME                 phr-3-30a
EXPNO                        1
PROCNO                       1
Date_             20081107
Time_                10.17
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         203
DW                    60.800 usec
DE                     6.50 usec
TE                    296.7 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.80 usec
PL1                    -1.00 dB
PL1W            13.18669796 W
SFO1            400.1724712 MHz
SI                       32768
SF              400.1700000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

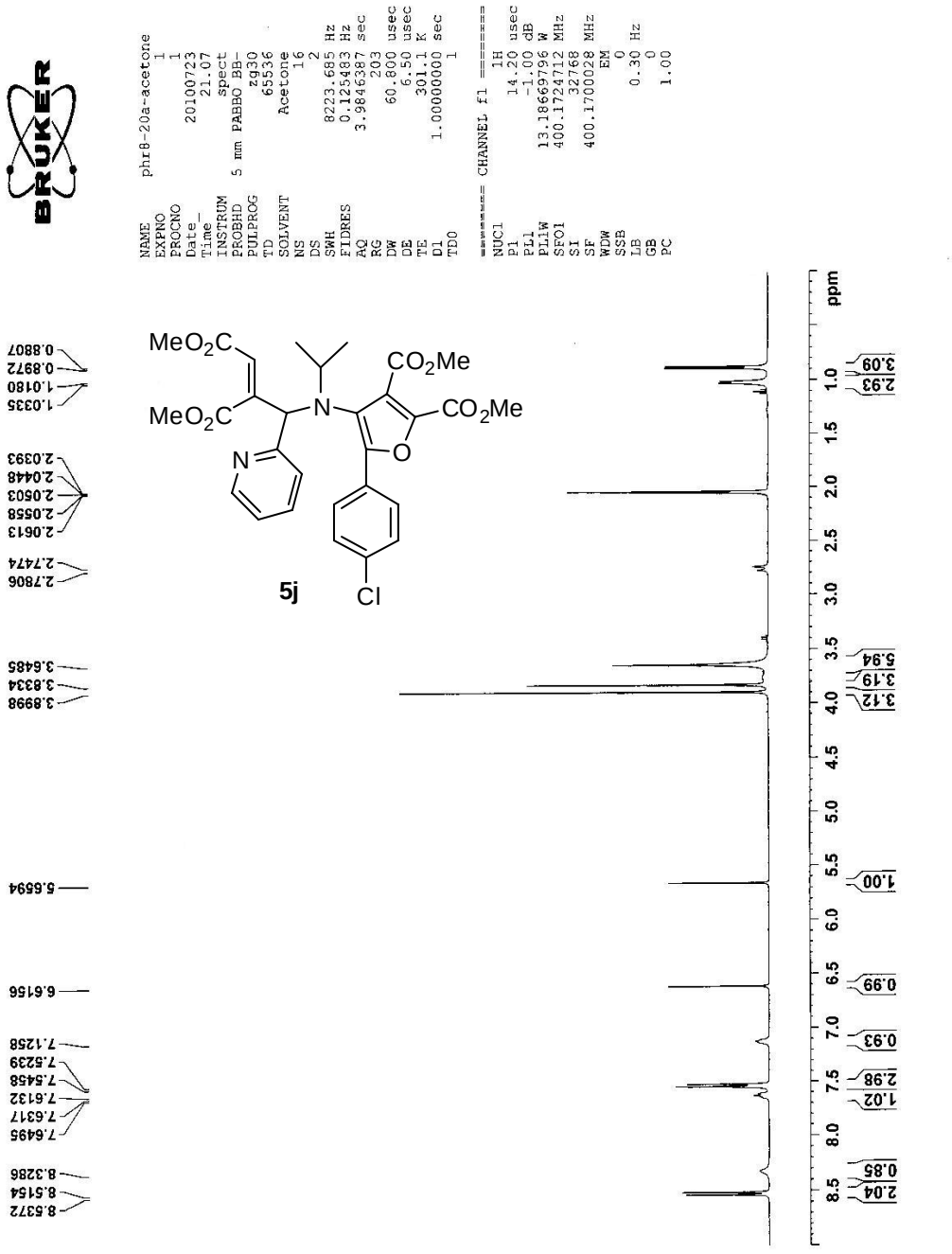





```
NAME                 phr-3-30a-c13
EXPNO                           1
PROCNO                          1
Date_                    20081121
Time_                       16.32
INSTRUM                      spect
PROBHD     5 mm PABBO BB-
PULPROG                  zgpg30
TD                            65536
SOLVENT                   CDCl3
NS                             4800
DS                                4
SWH                      24038.461 Hz
FIDRES                    0.366798 Hz
AQ                        1.3631988 sec
RG                              203
DW                         20.800 usec
DE                          6.50 usec
TE                           298.1 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                           13C
P1                            8.50 usec
PL1                          -2.00 dB
PL1W                    57.32743073 W
SFO1                   100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                      80.00 usec
PL2                         -1.00 dB
PL12                        14.26 dB
PL13                        14.46 dB
PL2W                    13.18669796 W
PL12W                    0.39276794 W
PL13W                    0.37509048 W
SFO2                   400.1716007 MHz
SI                            32768
SF                     100.6228270 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           0.05
```


NAME phr8-20a-acetone
EXPNO 1
PROCNO 1
Date_ 20100723
Time_ 21.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 301.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO2C
CO2Me
MeO2C
N
CO2Me
N
O
Cl
5j
0.8807
0.8972
1.0180
1.0335
2.0393
2.0448
2.0603
2.0558
2.0613
2.7474
2.7806
3.6485
3.8334
3.8998
5.6594
6.6156
7.1258
7.5239
7.5458
7.6132
7.6317
7.6495
8.3286
8.5154
8.5372
3.09
2.93
5.94
3.19
3.12
1.00
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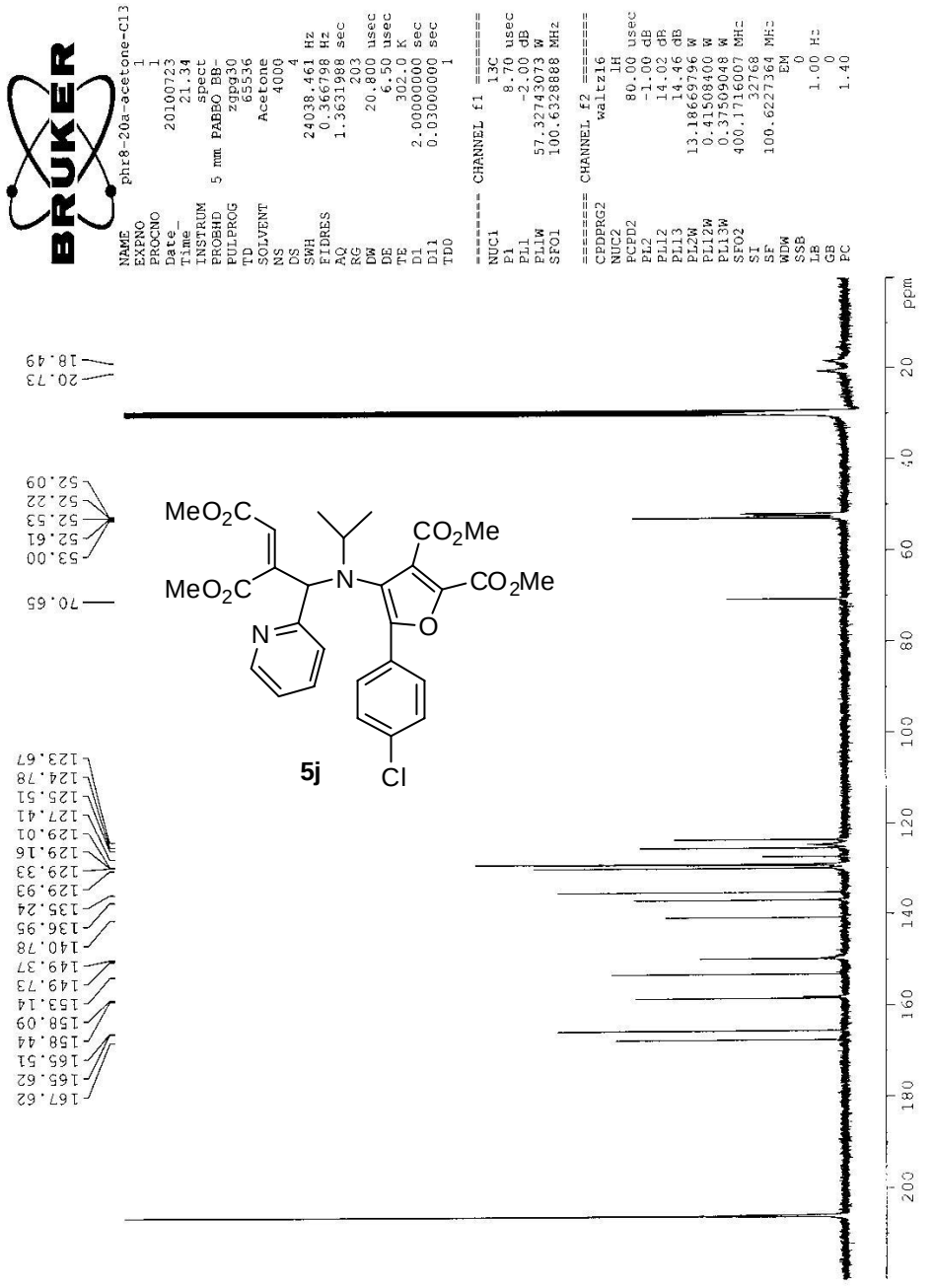
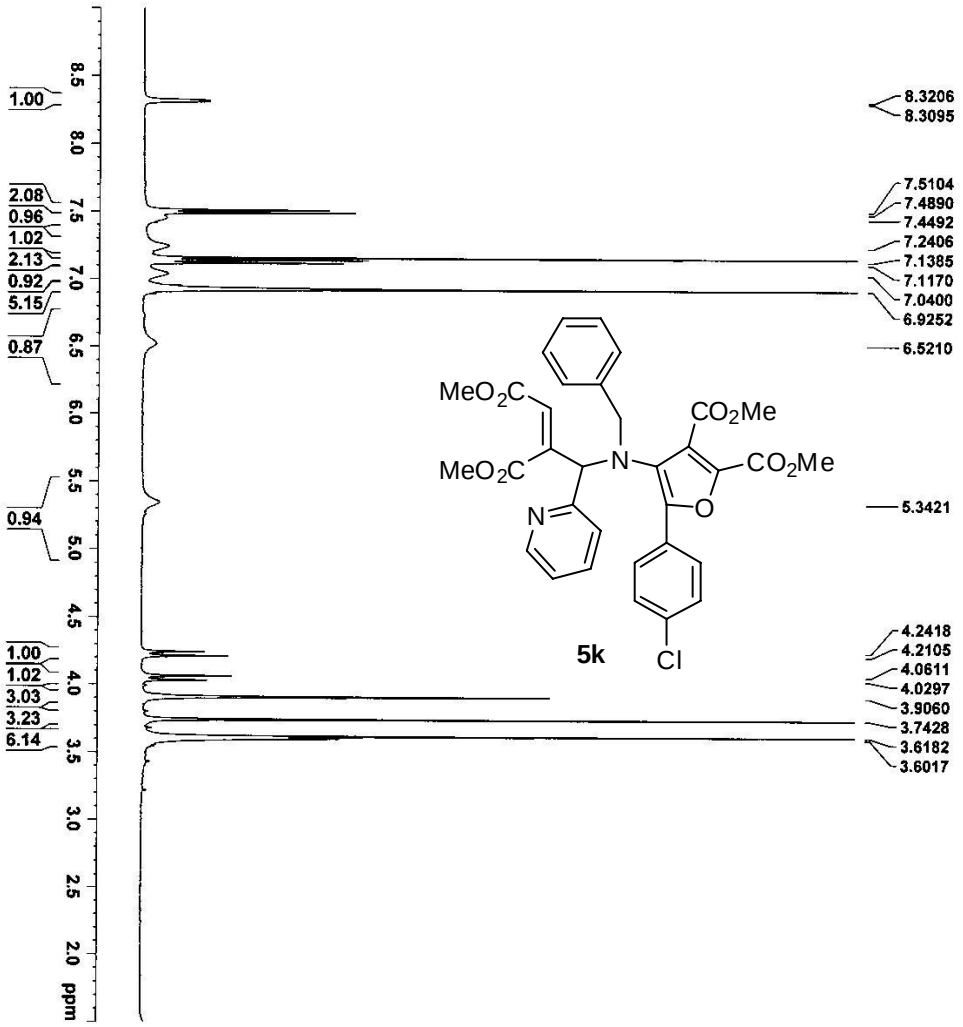
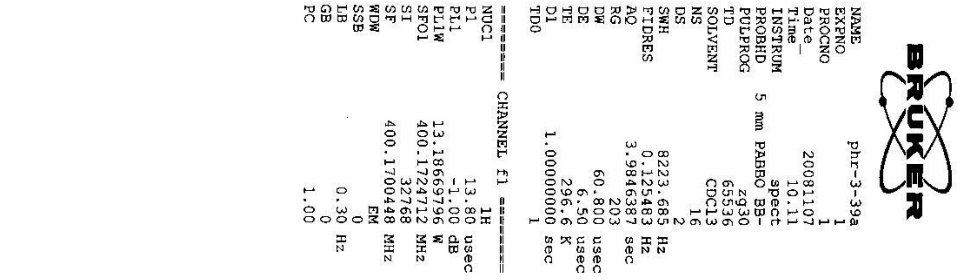
0.93
2.98
1.02
0.85
2.04
ppm

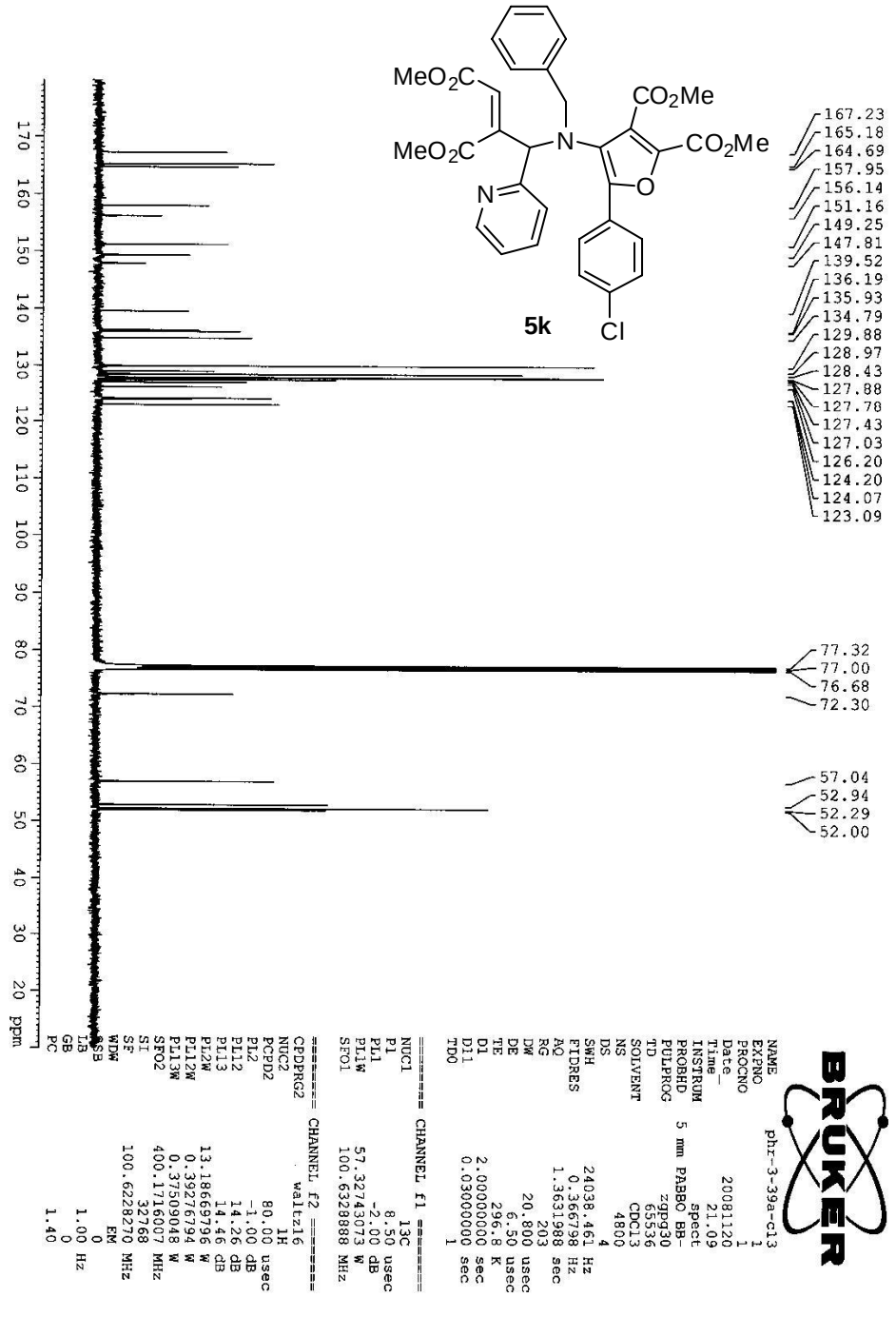

NAME phr8-20a-acetone-C13
EXPNO 1
PROCNO 1
Date_ 20100723
Time_ 21.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 4000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 302.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6227364 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO2C
MeO2C
N
CO2Me
CO2Me
O
N
Cl
5j
18.49
20.73
52.09
52.22
52.53
52.61
53.00
70.65
123.67
124.78
125.51
127.41
129.01
129.16
129.33
129.93
135.24
136.95
140.78
149.37
149.73
153.14
158.09
158.44
165.51
165.62
167.62
ppm
200
180
160
140
120
100
80
60
40
20

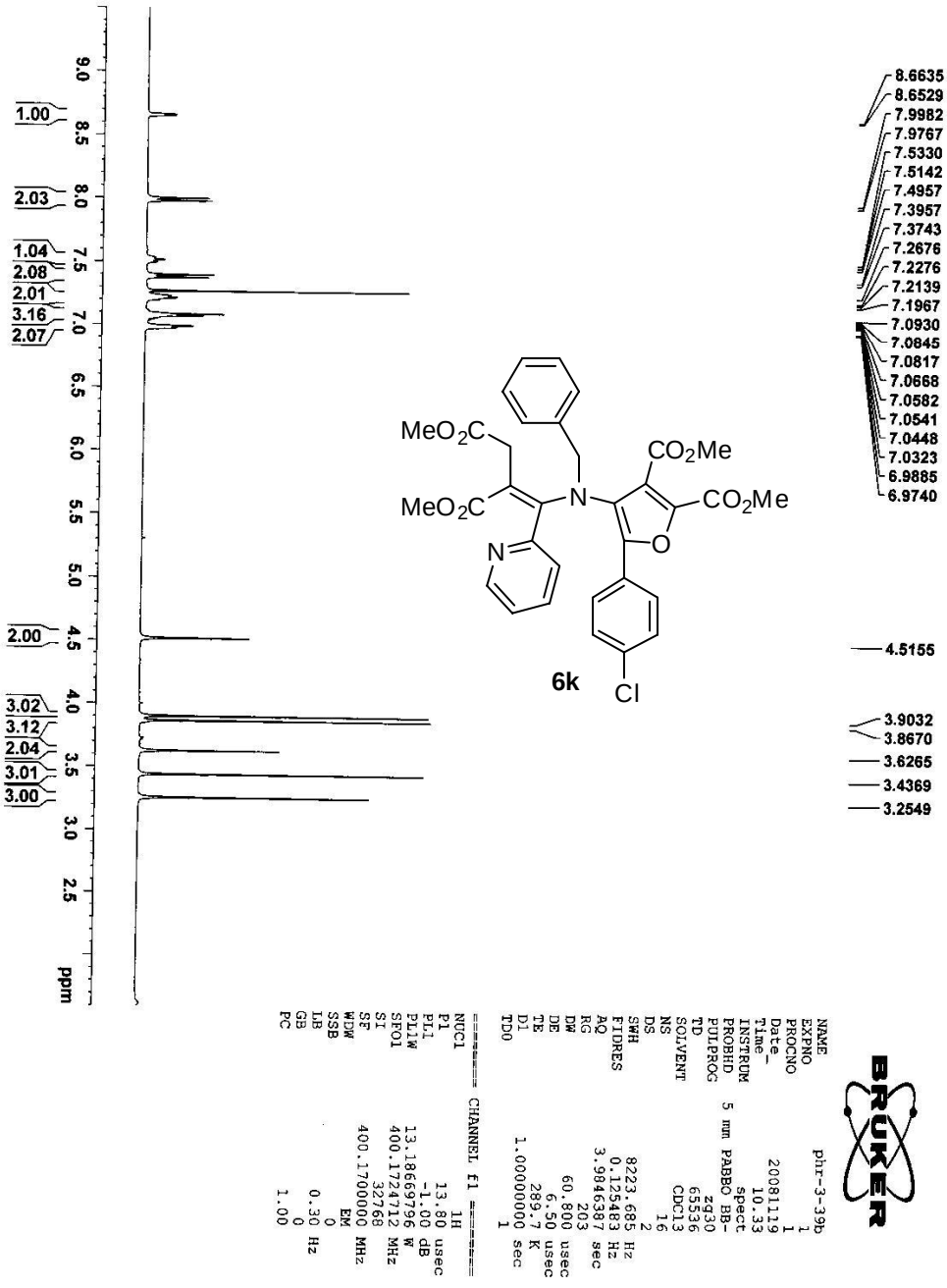




```
NAME                 phr-3-39a
EXPNO                        1
PROCNO                       1
Date_             20081107
Time_                    10.11
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       203
DW                  60.800 usec
DE                   6.50 usec
TE                   296.6 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                  13.80 usec
PL1                  -1.00 dB
PLIW                13.18669796 W
SFO1               400.1724712 MHz
SI                       32768
SF                 400.1700448 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00
```


MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
5k
167.23
165.18
164.69
157.95
156.14
151.16
149.25
147.81
139.52
136.19
135.93
134.79
129.88
128.97
128.43
127.88
127.78
127.43
127.03
126.20
124.20
124.07
123.09
77.32
77.00
76.68
72.30
57.04
52.94
52.29
52.00
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
BRUKER
phr-3-39a-c13
NAME
EXPNO 1
PROCNO 1
Date_ 20081120
Time 21.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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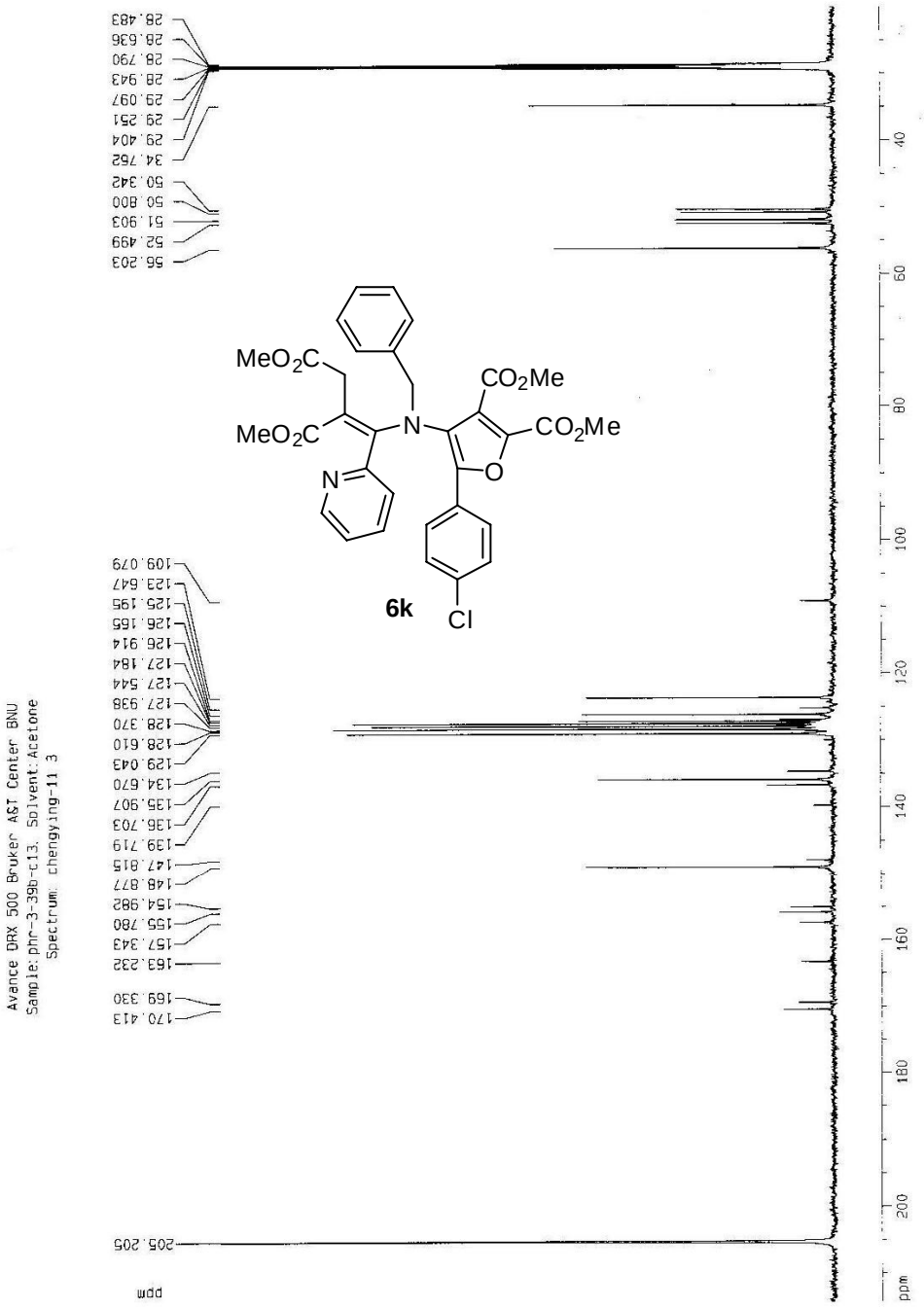
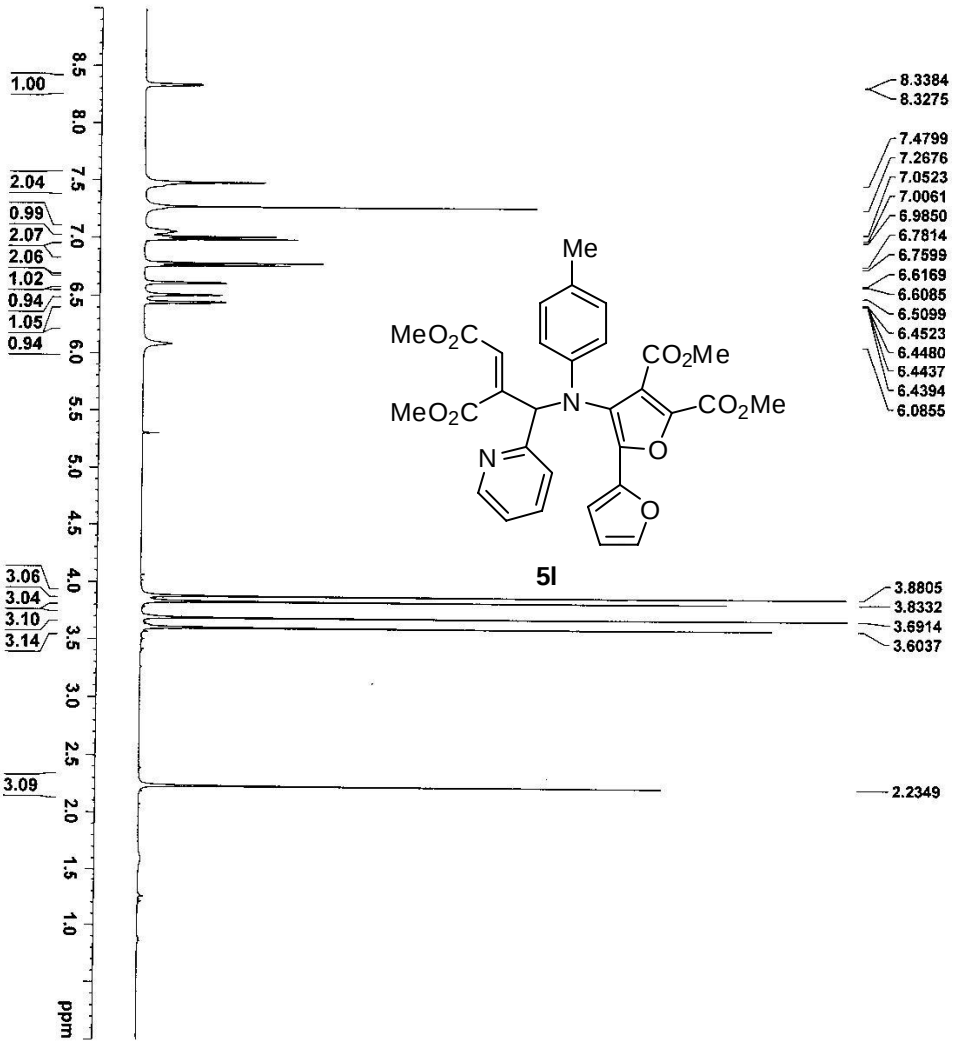
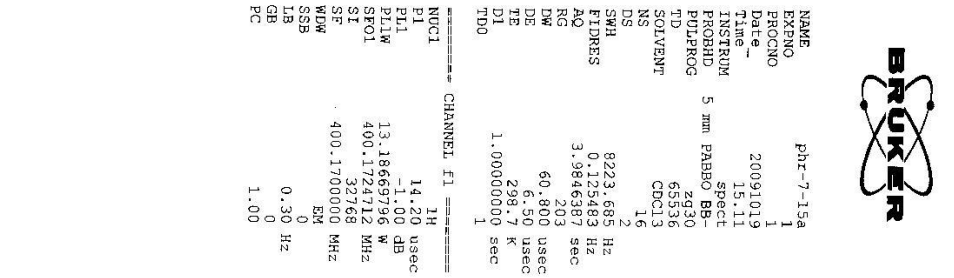
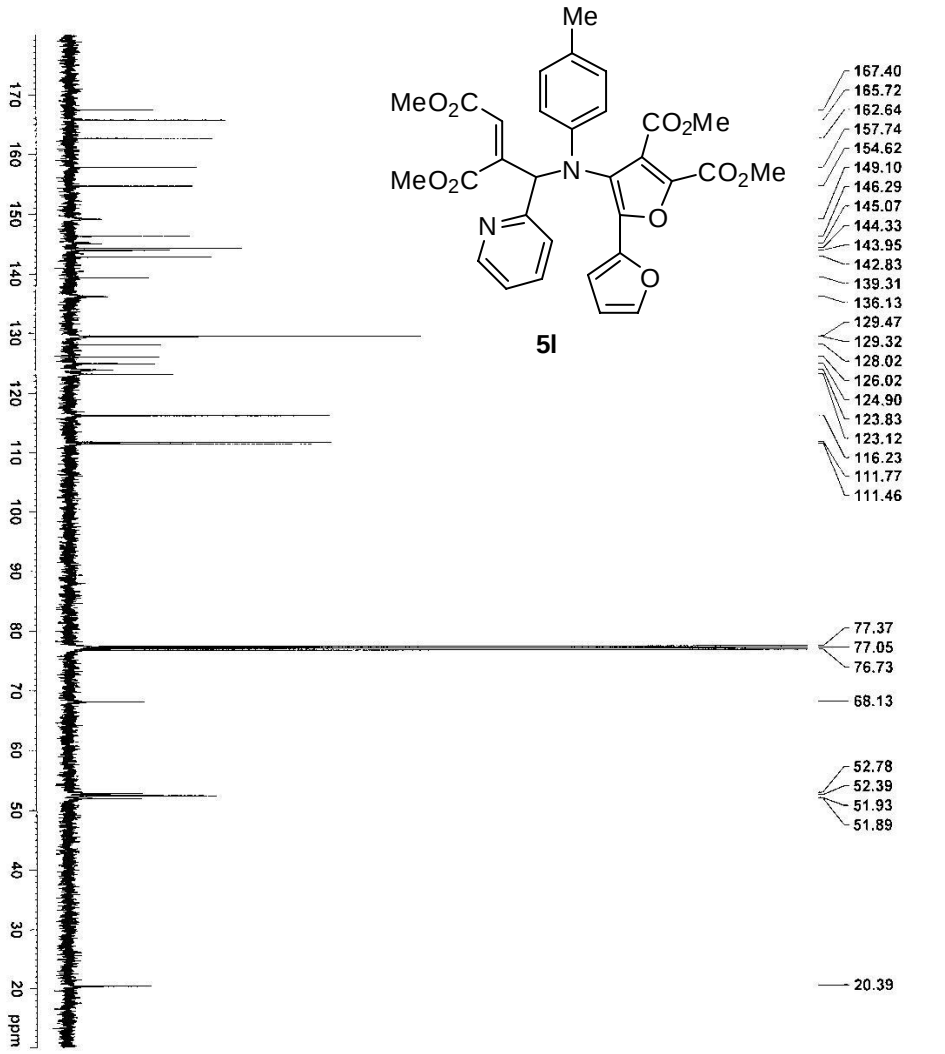
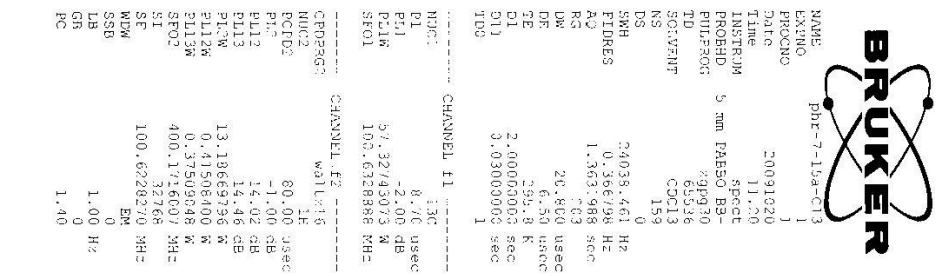
8.6529
7.9982
7.9767
7.5330
7.5142
7.4957
7.3957
7.3743
7.2676
7.2276
7.2139
7.1967
7.0930
7.0845
7.0817
7.0668
7.0582
7.0541
7.0448
7.0323
6.9885
6.9740
4.5155
3.9032
3.8670
3.6265
3.4369
3.2549
1.00
2.03
1.04
2.08
2.01
3.16
2.07
2.00
3.02
3.12
2.04
3.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
MeO2C
MeO2C
CO2Me
CO2Me
N
O
N
Cl
6k
NAME phr-3-39b
EXPNO 1
PROCNO 1
Date_ 20081119
Time_ 10.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 289.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER

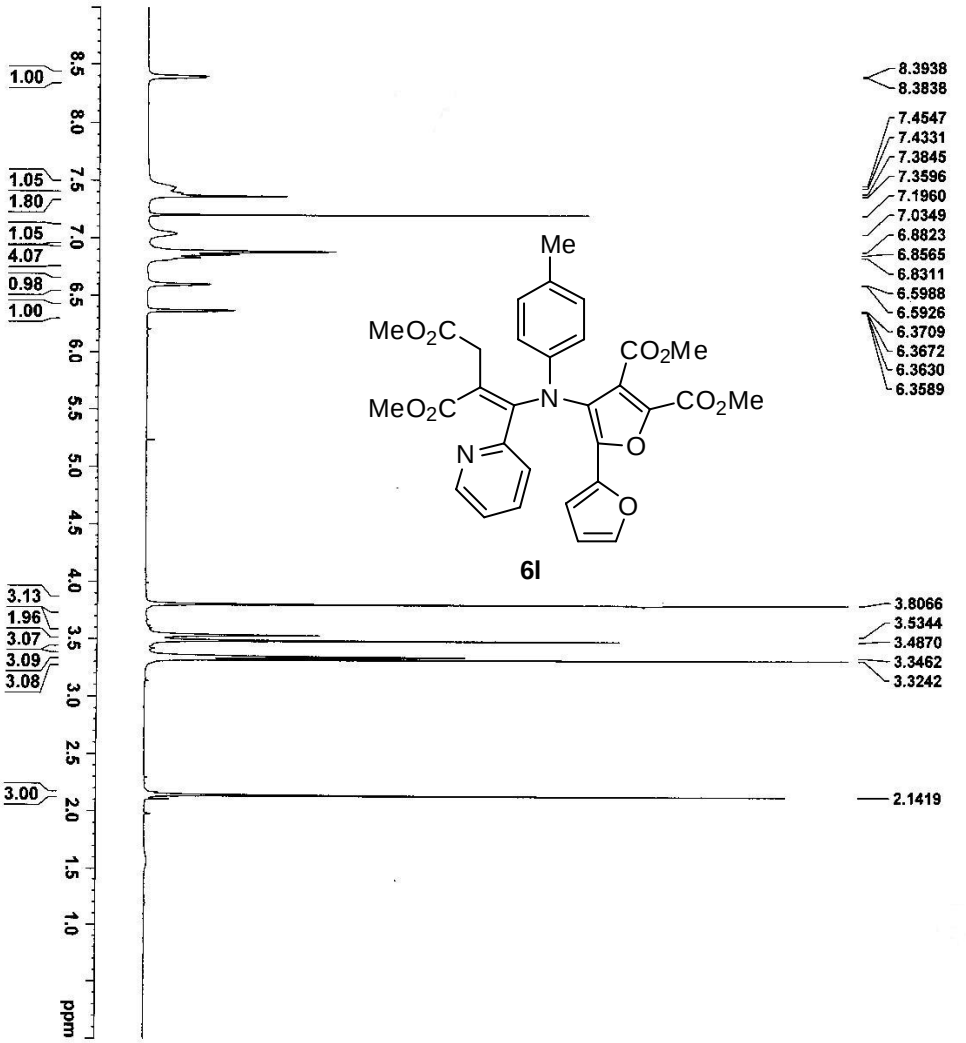

Avance DRX 500 Bruker AST Center BNU
Sample: phr-3-39b-c13, Solvent: Acetone
Spectrum: chengying-11 3
28.483
28.636
28.790
28.943
29.097
29.251
29.404
34.752
50.342
50.800
51.903
52.499
56.203
109.079
123.647
125.195
126.165
126.914
127.184
127.544
127.938
128.370
128.610
129.043
134.670
135.907
136.703
139.719
147.815
148.877
154.982
155.780
157.343
163.232
169.330
170.413
205.205
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
Cl
6k
ppm
40
60
80
100
120
140
160
180
200
ppm

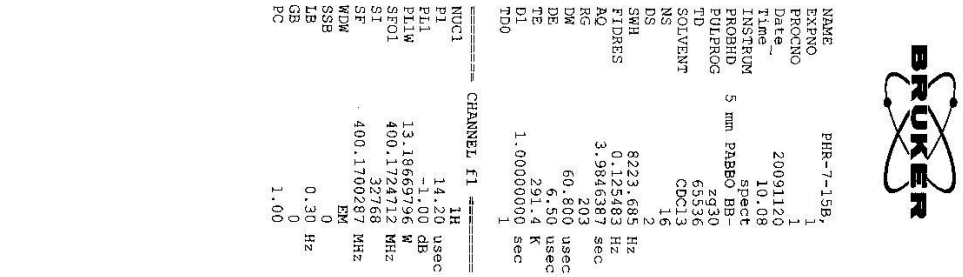

8.3384
8.3275
7.4799
7.2676
7.0523
7.0061
6.9850
6.7814
6.7599
6.6169
6.6085
6.5099
6.4523
6.4480
6.4437
6.4394
6.0855
3.8805
3.8332
3.6914
3.6037
2.2349
1.00
2.04
0.99
2.07
2.06
1.02
0.94
1.05
0.94
3.06
3.04
3.10
3.14
3.09
ppm
Me
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
O
5l

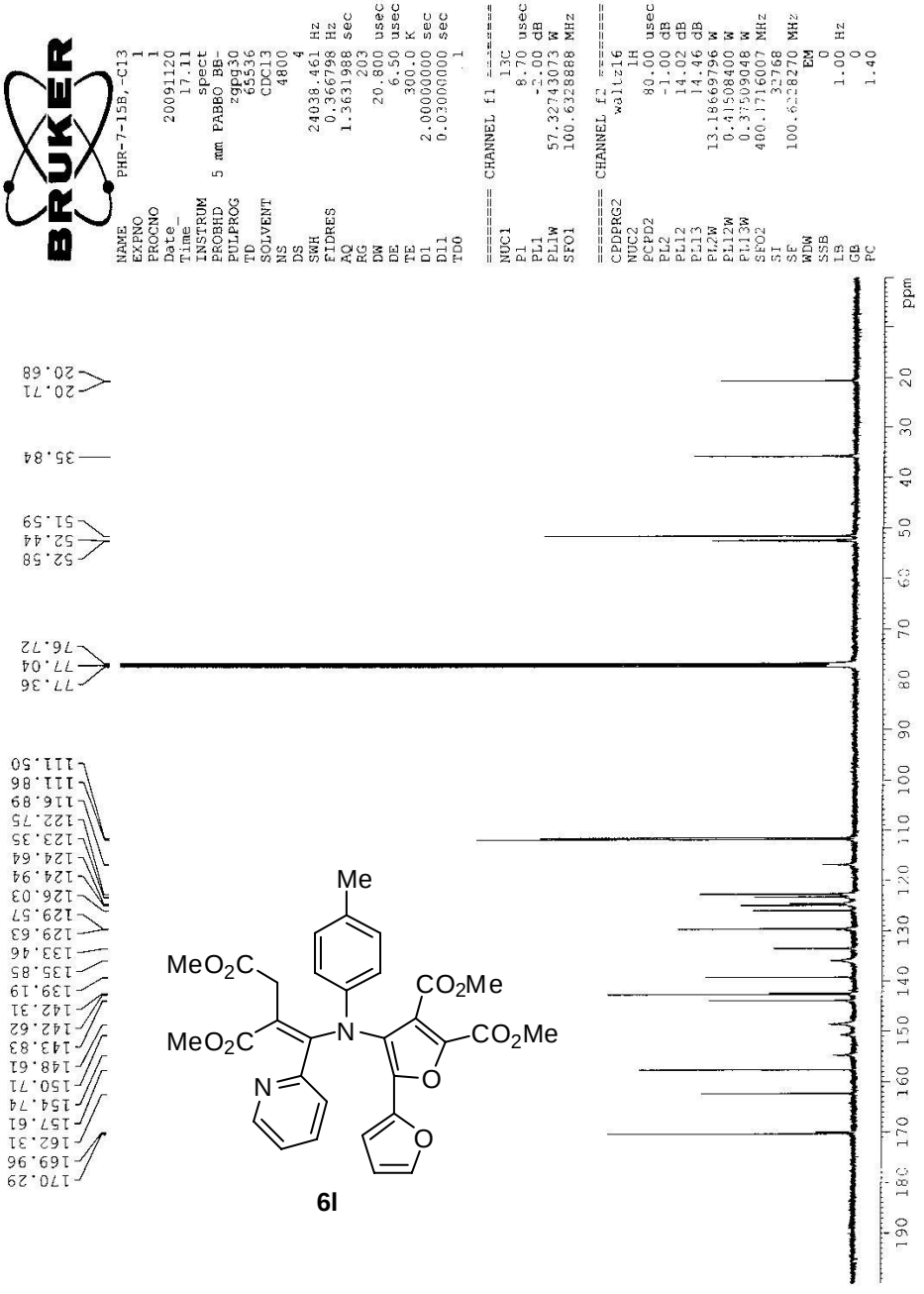

NAME phr-7-15a
EXPNO 1
PROCNO 1
Date_ 20091019
Time_ 15.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


Me
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
O
5l
167.40
165.72
162.64
157.74
154.62
149.10
146.29
145.07
144.33
143.95
142.83
139.31
136.13
129.47
129.32
128.02
126.02
124.90
123.83
123.12
116.23
111.77
111.46
77.37
77.05
76.73
68.13
52.78
52.39
51.93
51.89
20.39
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

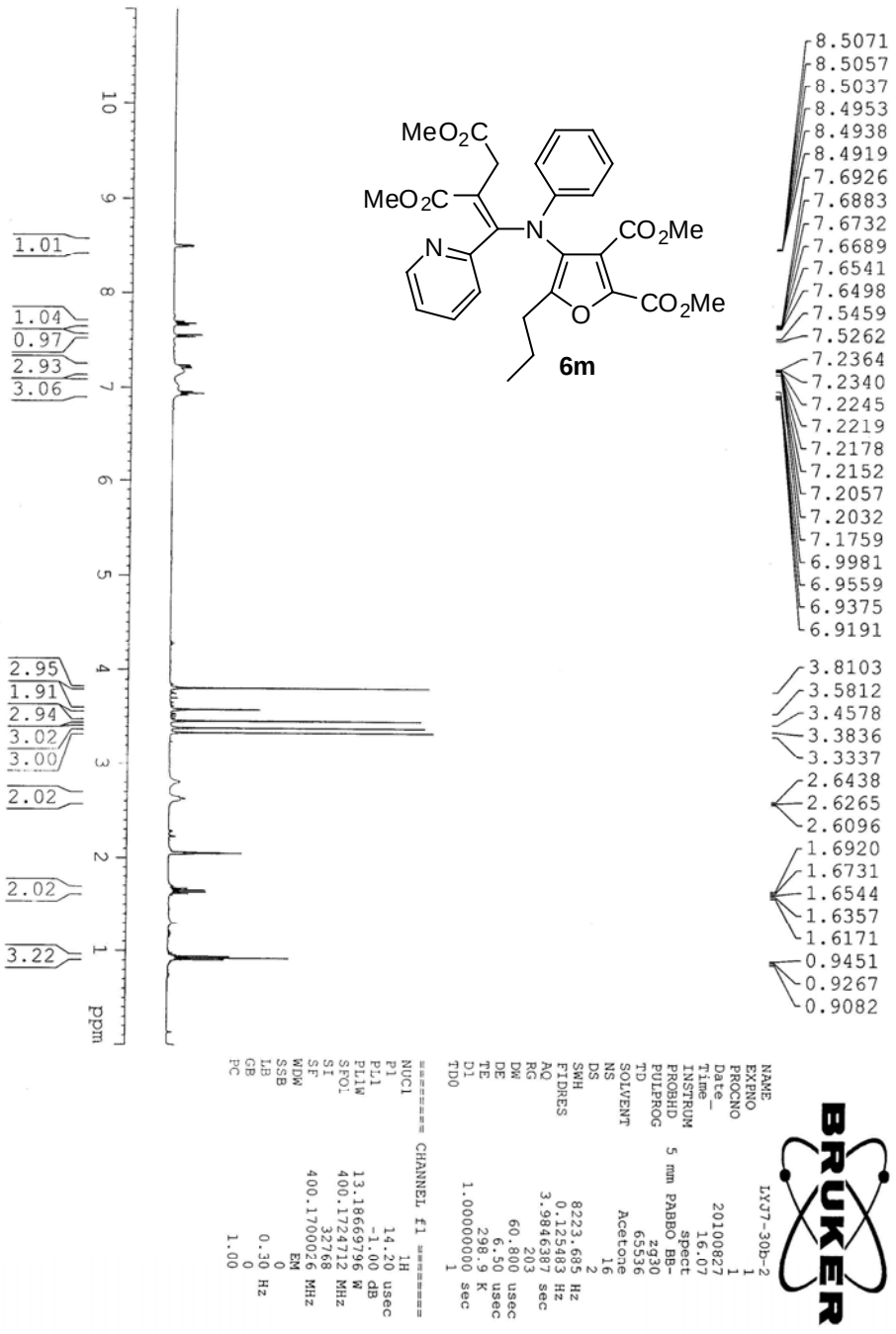

NAME phr-7-15a-C13
EXPNO 1
PROCNO 1
Date 20091020
Time 11.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 159
DS 0
SWH 24038.461 Hz
FIDRES 0.366998 Hz
AQ 1.363988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000003 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6228886 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL1W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1316007 MHz
SI 32768
SF 100.6220270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

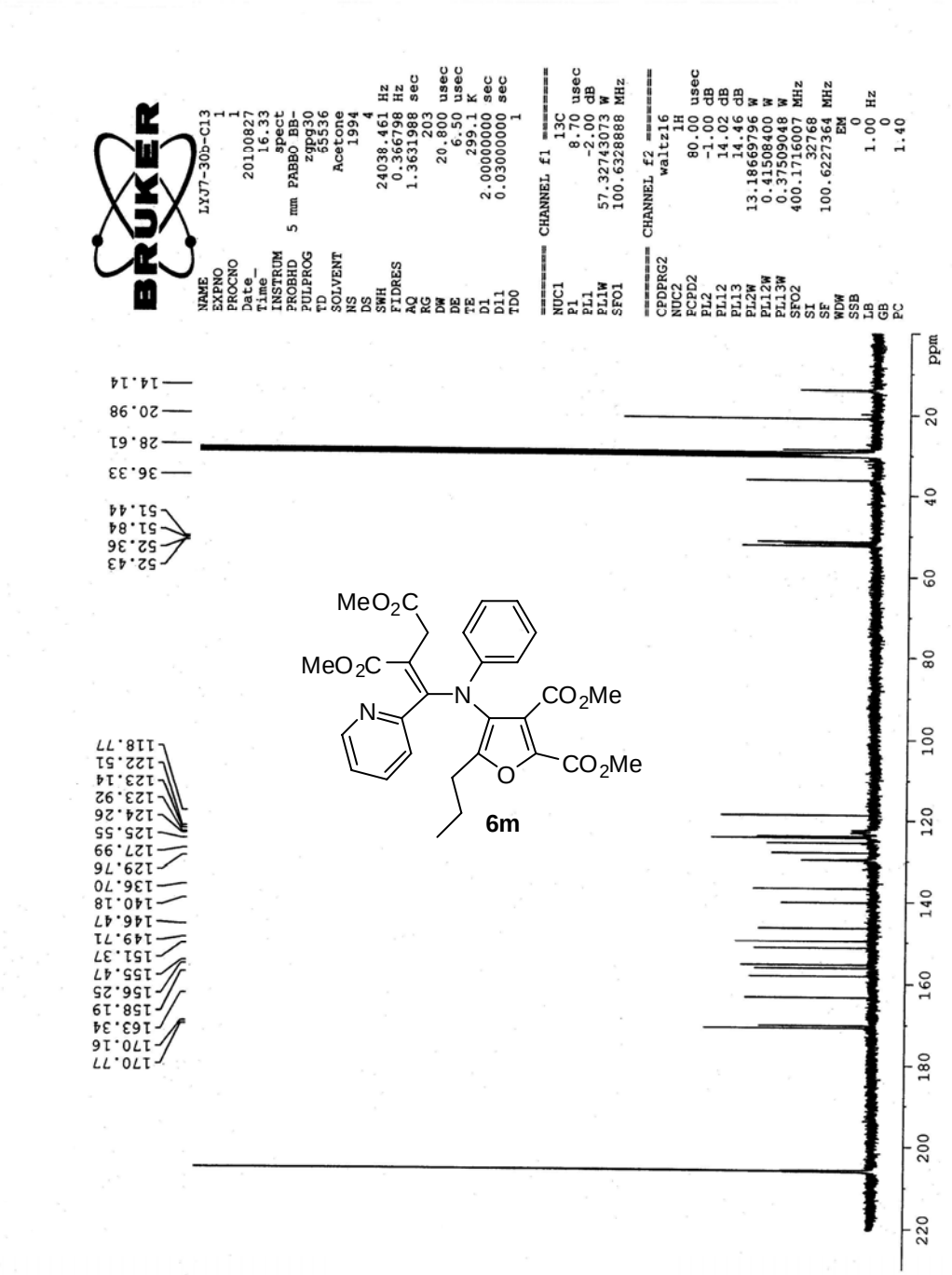

8.3938
8.3838
7.4547
7.4331
7.3845
7.3596
7.1960
7.0349
6.8823
6.8565
6.8311
6.5988
6.5926
6.3709
6.3672
6.3630
6.3589
3.8066
3.5344
3.4870
3.3462
3.3242
2.1419
1.00
1.05
1.80
1.05
4.07
0.98
1.00
3.13
1.96
3.07
3.09
3.08
3.00
ppm
Me
MeO2C
MeO2C
CO2Me
CO2Me
N
N
O
O
6l


NAME PHR-7-15B,
EXPNO 1
PROCNO 1
Date_ 20091120
Time 10.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 291.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1324712 MHz
SI 32768
SF 400.1700287 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


BRUKER
NAME PHR-7-15B,-C13
EXPNO 1
PROCNO 1
Date_ 20091120
Time_ 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
20.68
20.71
35.84
51.59
52.44
52.58
76.72
77.04
77.36
111.50
111.86
116.89
122.75
123.35
124.64
124.94
126.03
129.57
129.63
133.46
135.85
139.19
142.31
142.62
143.83
148.61
150.71
154.74
157.61
162.31
169.96
170.29
Me
MeO2C
MeO2C
N
CO2Me
CO2Me
O
N
O

6l


MeO2C
MeO2C
N
N
CO2Me
CO2Me
O
6m
8.5071
8.5057
8.5037
8.4953
8.4938
8.4919
7.6926
7.6883
7.6732
7.6689
7.6541
7.6498
7.5459
7.5262
7.2364
7.2340
7.2245
7.2219
7.2178
7.2152
7.2057
7.2032
7.1759
6.9981
6.9559
6.9375
6.9191
3.8103
3.5812
3.4578
3.3836
3.3337
2.6438
2.6265
2.6096
1.6920
1.6731
1.6544
1.6357
1.6171
0.9451
0.9267
0.9082
1.01
1.04
0.97
2.93
3.06
2.95
1.91
2.94
3.02
3.00
2.02
2.02
3.22
ppm
NAME LXJ7-30b-2
EXPNO 1
PROCNO 1
Date_ 20100827
Time 16.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


BRUKER
NAME LXJ7-30b-C13
EXPNO 1
PROCNO 1
Date_ 20100827
Time_ 16.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1994
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6227364 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
14.14
20.98
28.61
36.33
51.44
51.84
52.36
52.43
118.77
122.51
123.14
123.92
124.26
125.55
127.99
129.76
136.70
140.18
146.47
149.71
151.37
155.47
156.25
158.19
163.34
170.16
170.77
MeO2C
MeO2C
N
N
CO2Me
CO2Me
O
6m
ppm
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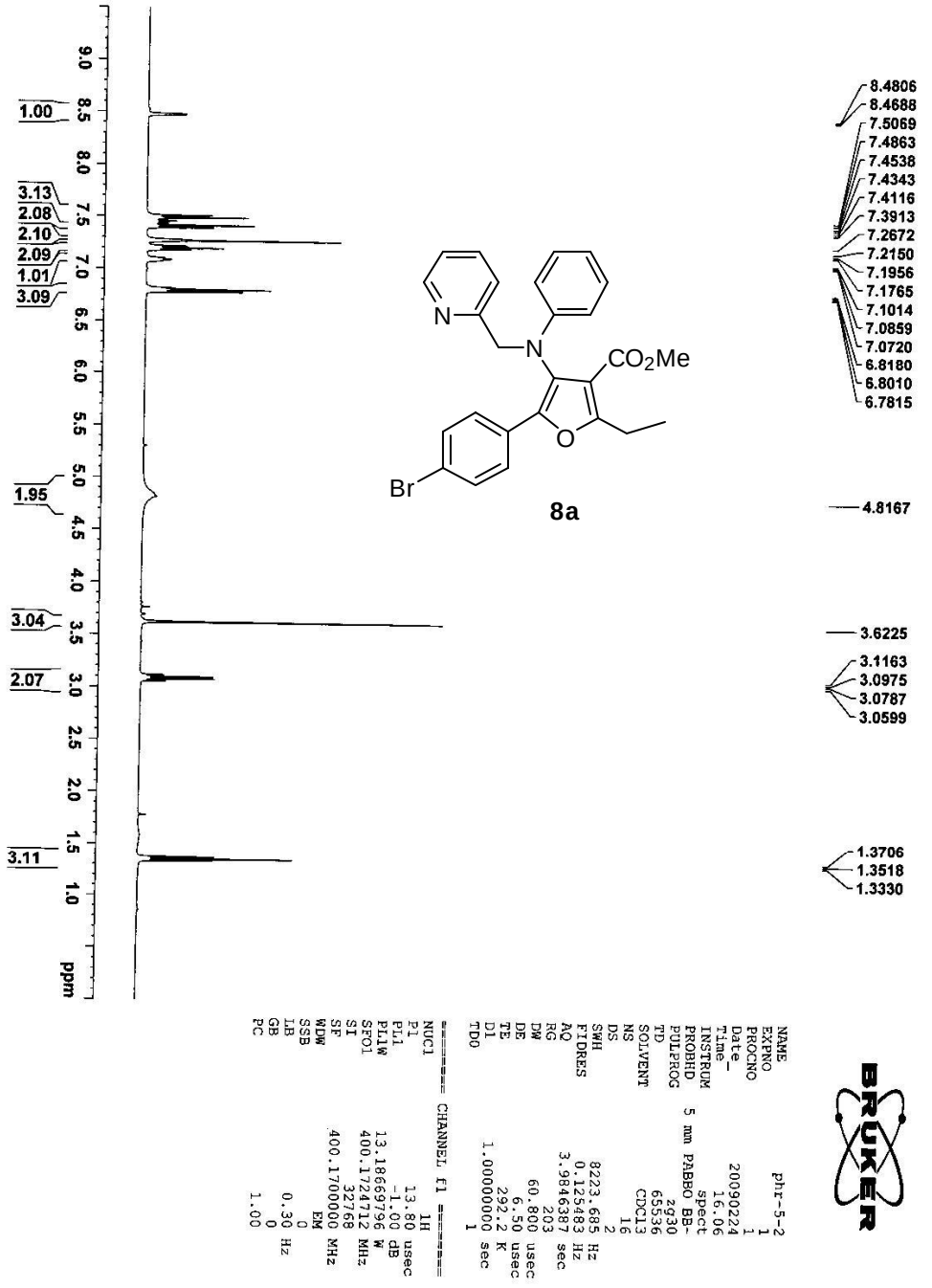
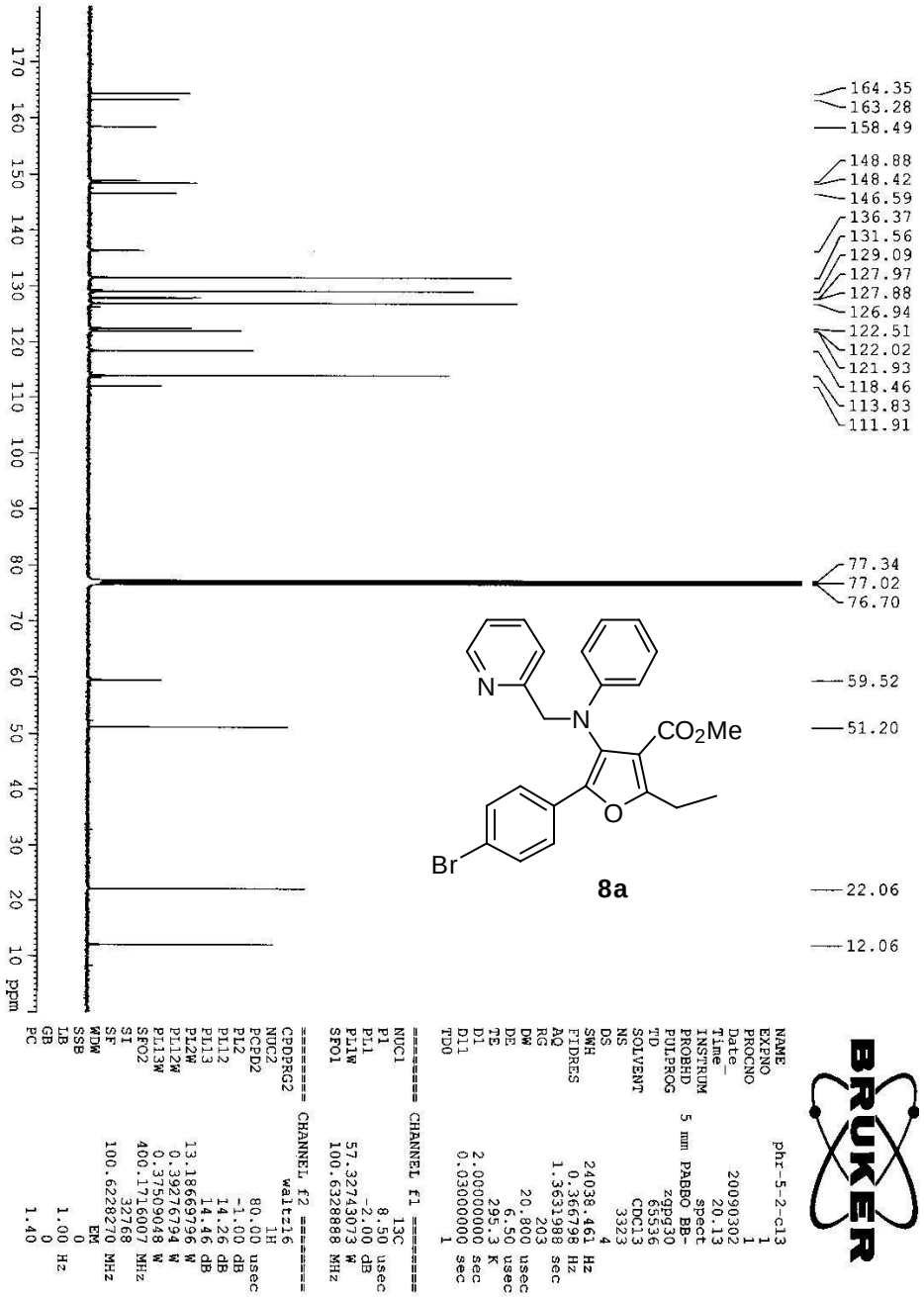
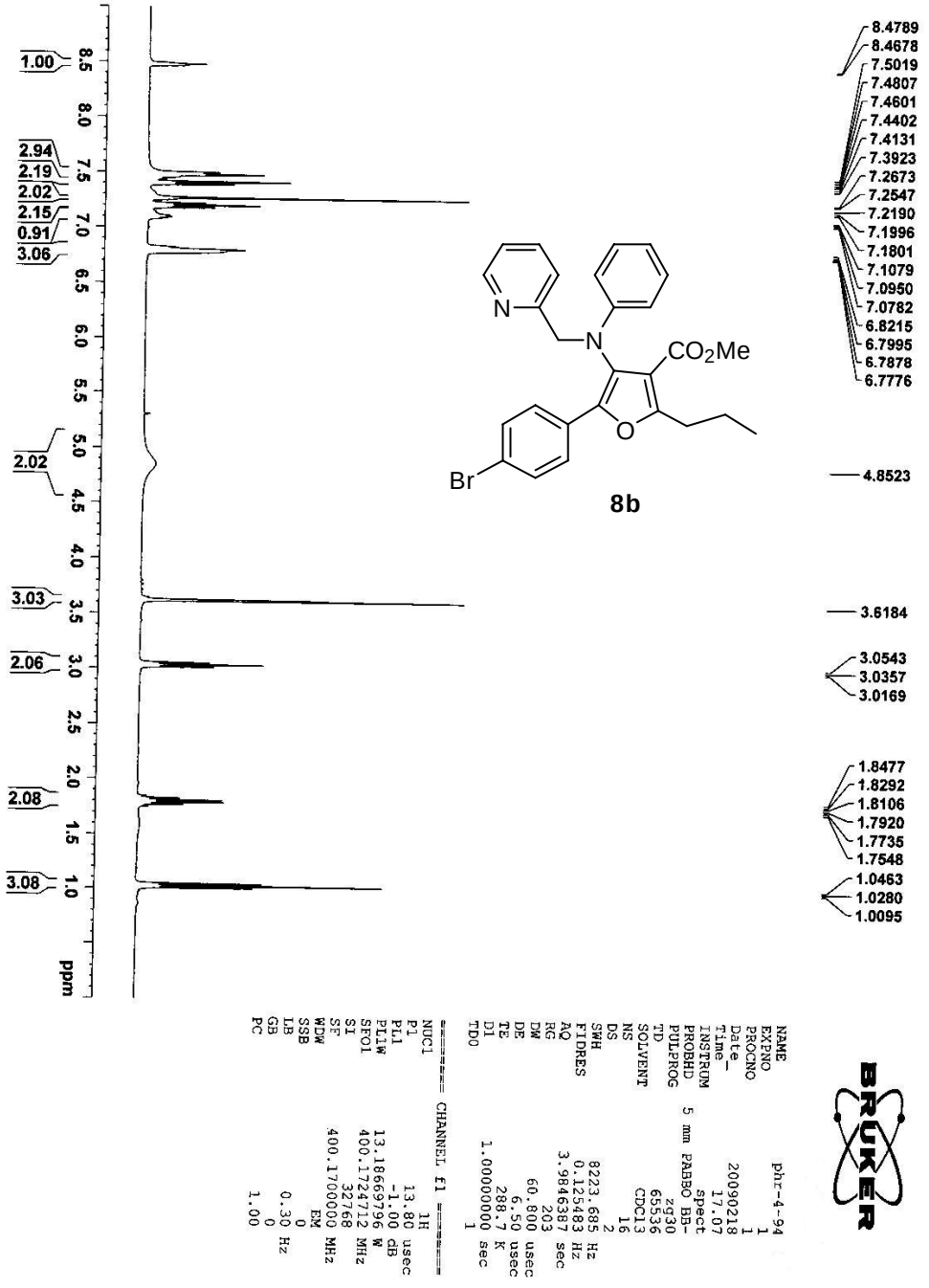
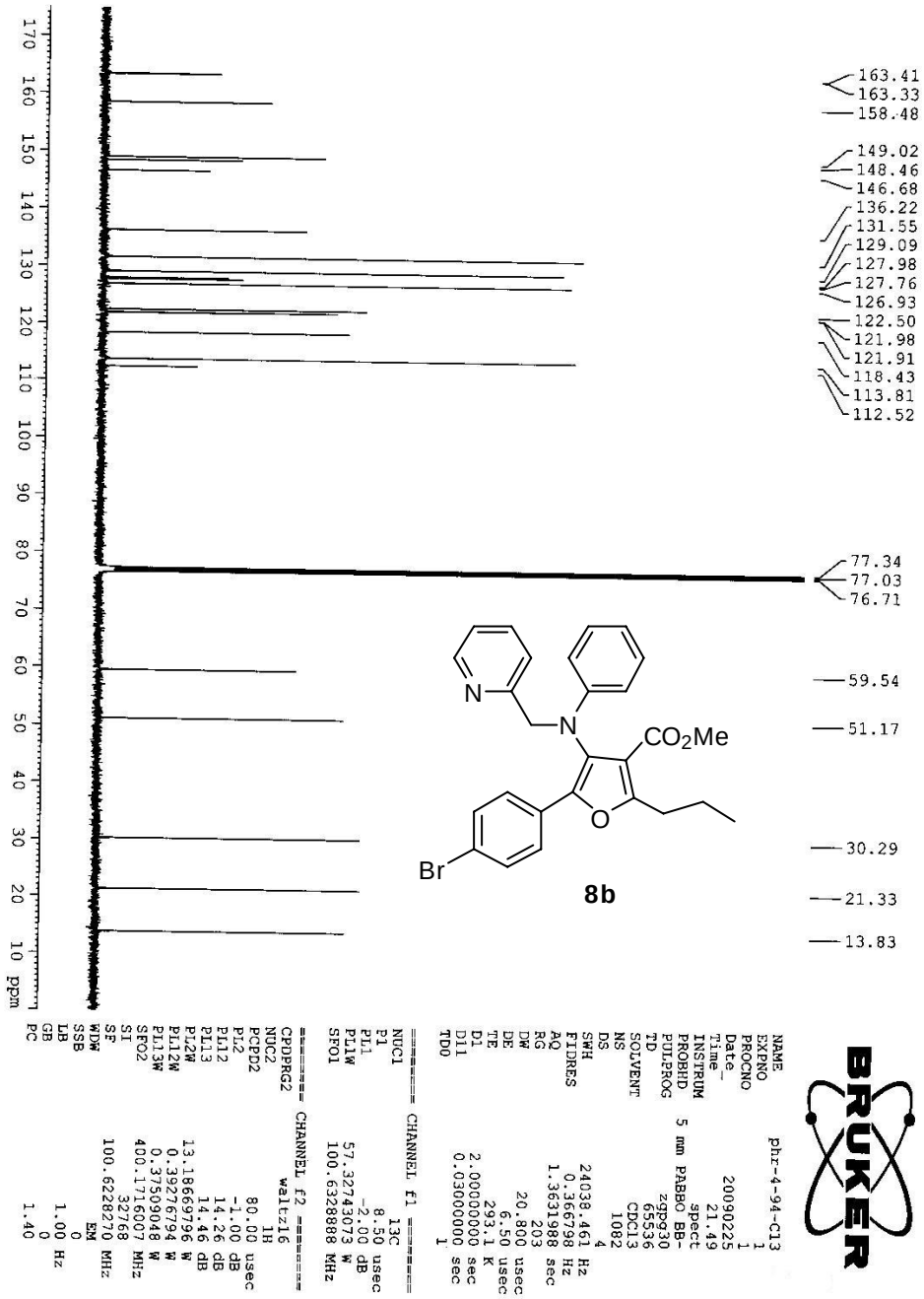
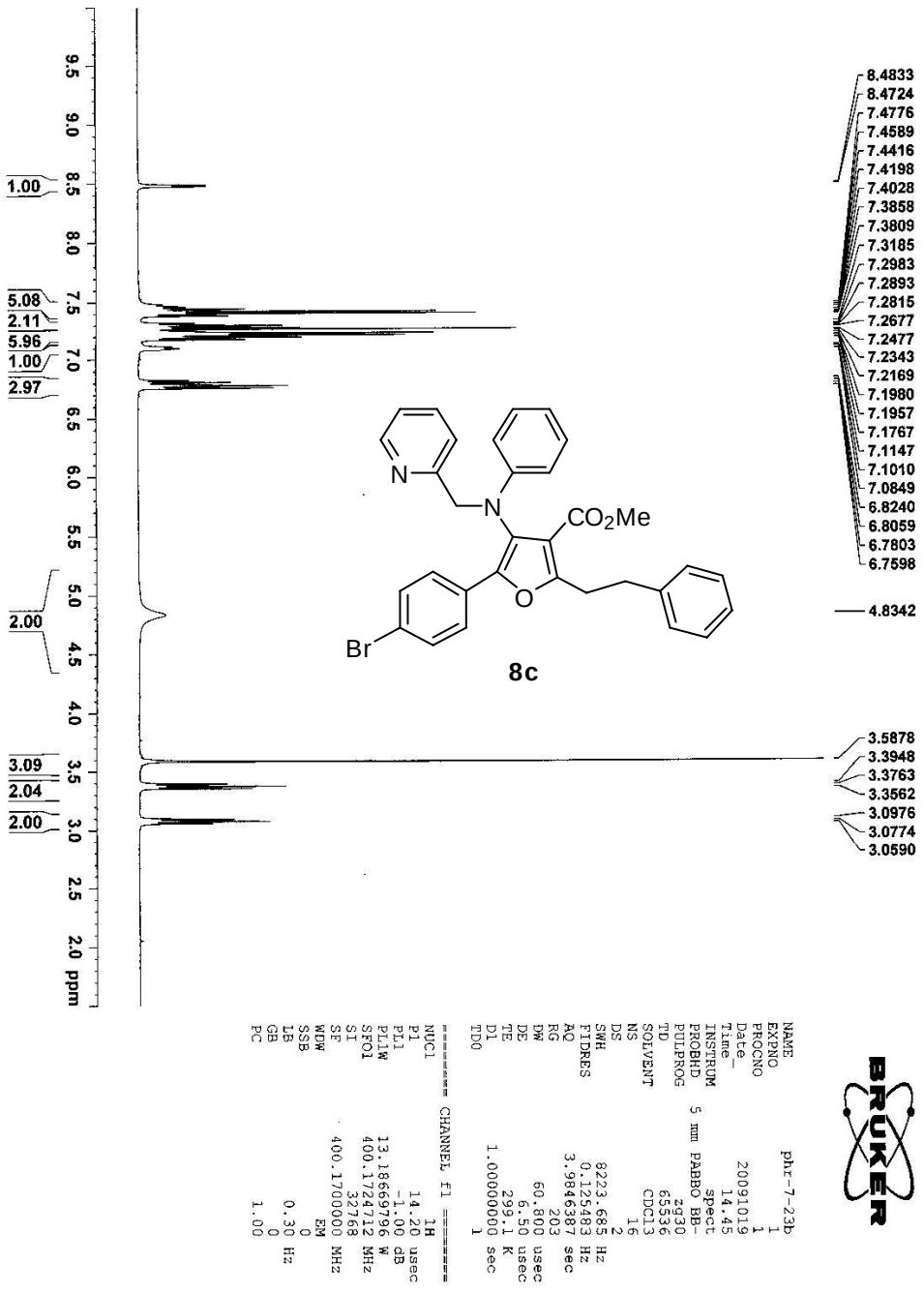
40
60
80
100
120
140
160
180
200
220


8.4806
8.4688
7.5069
7.4863
7.4538
7.4343
7.4116
7.3913
7.2672
7.2150
7.1956
7.1765
7.1014
7.0859
7.0720
6.8180
6.8010
6.7815
4.8167
3.6225
3.1163
3.0975
3.0787
3.0599
1.3706
1.3518
1.3330
1.00
3.13
2.08
2.10
2.09
1.01
3.09
1.95
3.04
2.07
3.11
ppm
N
N
CO2Me
O
Br
8a
NAME phr-5-2
EXPNO 1
PROCNO 1
Date_ 20090224
Time_ 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLIW 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


164.35
163.28
158.49
148.88
148.42
146.59
136.37
131.56
129.09
127.97
127.88
126.94
122.51
122.02
121.93
118.46
113.83
111.91
77.34
77.02
76.70
59.52
51.20
22.06
12.06
N
N
CO2Me
O
Br
8a
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
NAME phr-5-2-c13
EXPNO 1
PROCNO 1
Date_ 20090302
Time_ 20.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3323
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.4789
8.4678
7.5019
7.4807
7.4601
7.4402
7.4131
7.3923
7.2673
7.2547
7.2190
7.1996
7.1801
7.1079
7.0950
7.0782
6.8215
6.7995
6.7878
6.7776
4.8523
3.6184
3.0543
3.0357
3.0169
1.8477
1.8292
1.8106
1.7920
1.7735
1.7548
1.0463
1.0280
1.0095
1.00
2.94
2.19
2.02
2.15
0.91
3.06
2.02
3.03
2.06
2.08
3.08
ppm
N
N
CO2Me
O
Br
8b
NAME phr-4-94
EXPNO 1
PROCNO 1
Date_ 20090218
Time_ 17.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 288.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLW1 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


163.41
163.33
158.48
149.02
148.46
146.68
136.22
131.55
129.09
127.98
127.76
126.93
122.50
121.98
121.91
118.43
113.81
112.52
77.34
77.03
76.71
59.54
51.17
30.29
21.33
13.83
N
N
CO2Me
O
Br
8b
ppm
NAME phr-4-94-C13
EXPNO 1
PROCNO 1
Date_ 20090225
Time_ 21.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1082
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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7.4416
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7.4028
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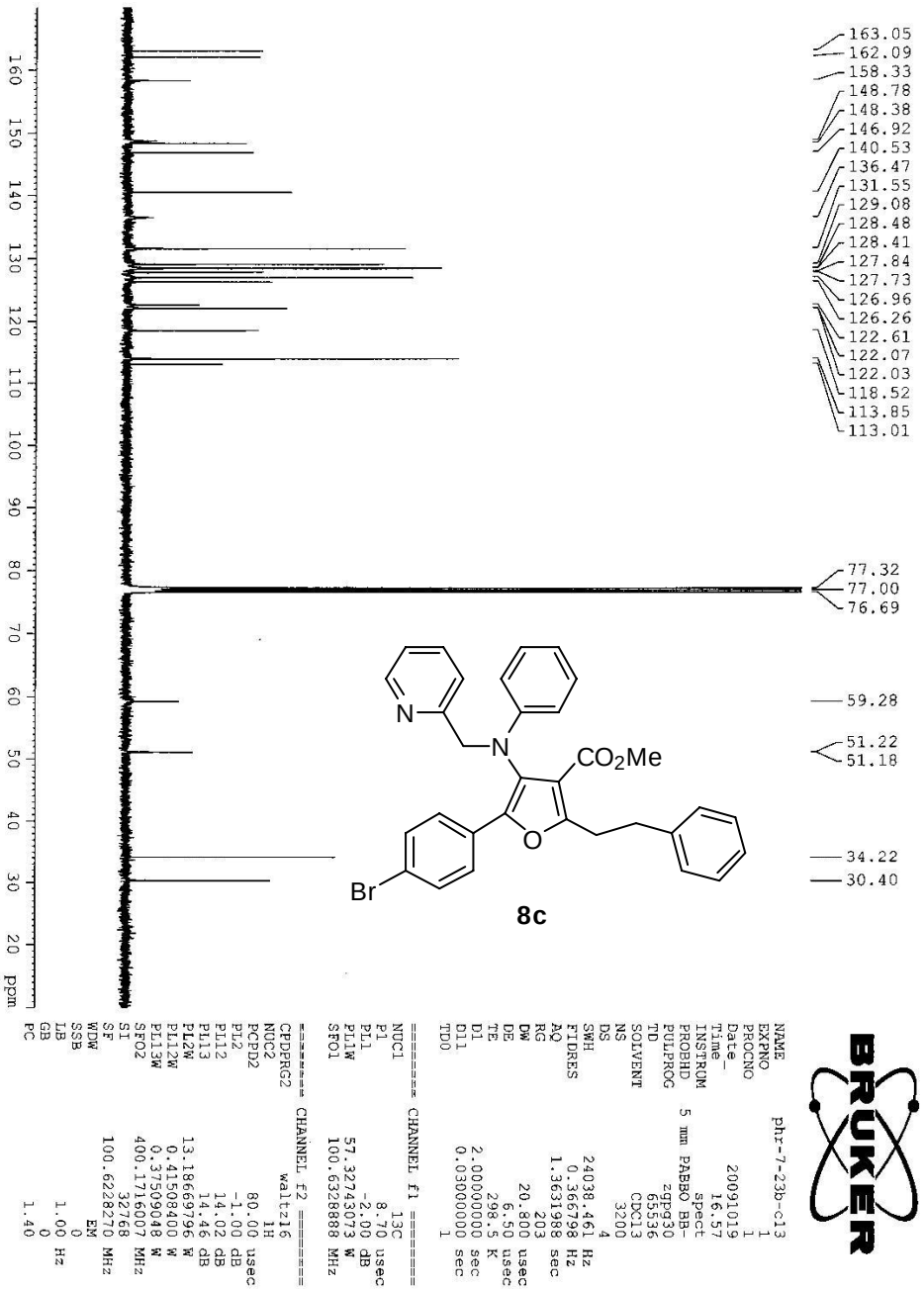
7.3809
7.3185
7.2983
7.2893
7.2815
7.2677
7.2477
7.2343
7.2169
7.1980
7.1957
7.1767
7.1147
7.1010
7.0849
6.8240
6.8059
6.7803
6.7598
4.8342
3.5878
3.3948
3.3763
3.3562
3.0976
3.0774
3.0590
1.00
5.08
2.11
5.96
1.00
2.97
2.00
3.09
2.04
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
N
N
CO2Me
O
Br
8c
NAME phr-7-23b
EXPNO 1
PROCNO 1
Date_ 20091019
Time_ 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER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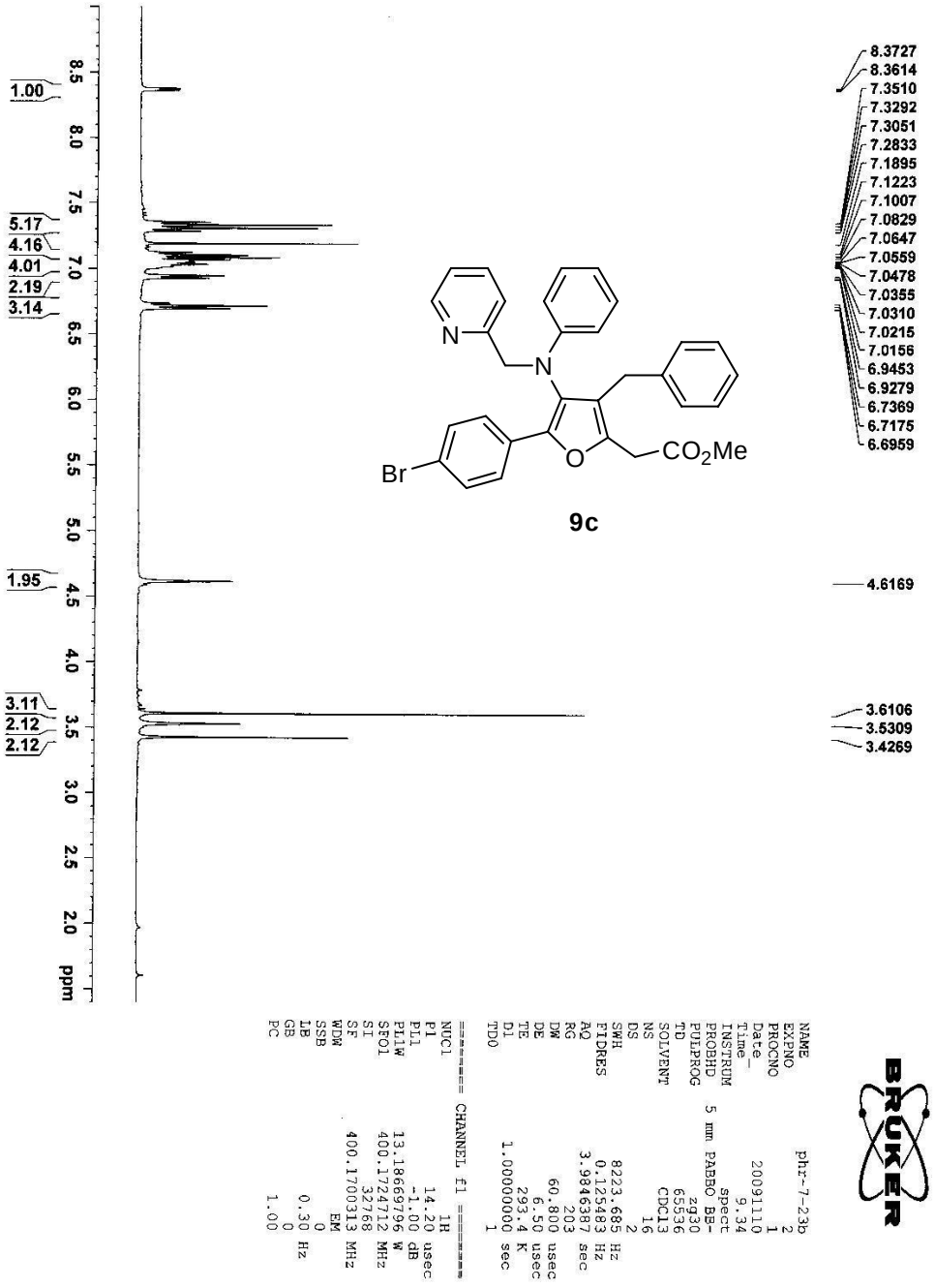
162.09
158.33
148.78
148.38
146.92
140.53
136.47
131.55
129.08
128.48
128.41
127.84
127.73
126.96
126.26
122.61
122.07
122.03
118.52
113.85
113.01
77.32
77.00
76.69
59.28
51.22
51.18
34.22
30.40
N
N
CO2Me
O
Br
8c
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
NAME phr-7-23b-c13
EXPNO 1
PROCNO 1
Date_ 20091019
Time_ 16.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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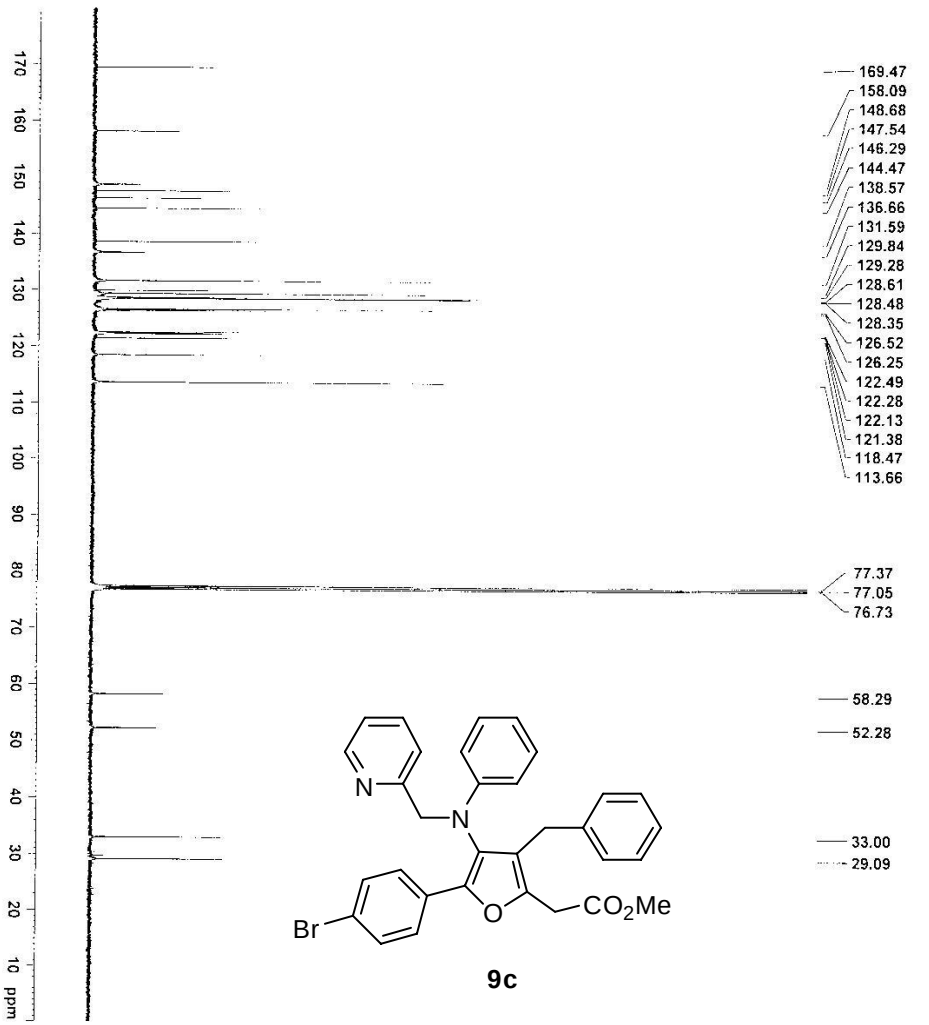
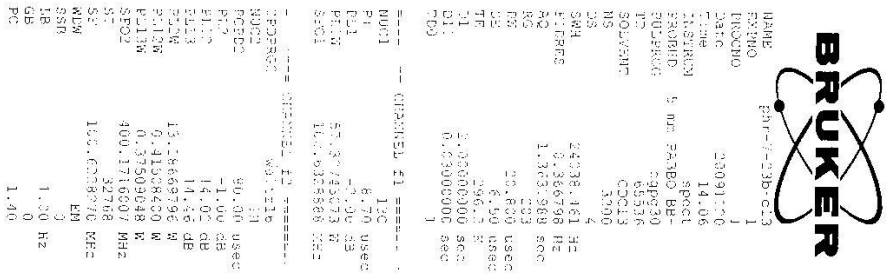
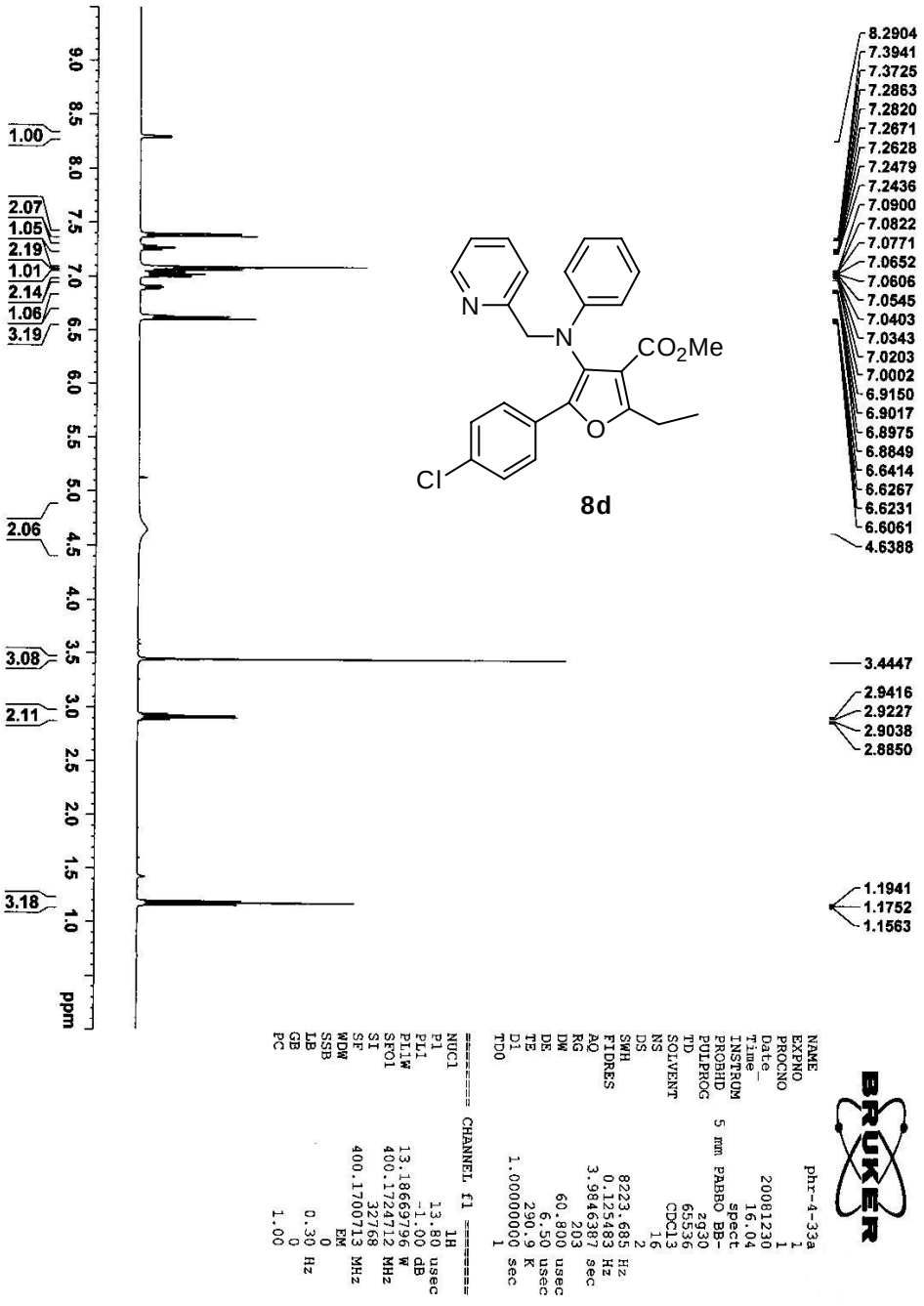
7.1007
7.0829
7.0647
7.0559
7.0478
7.0355
7.0310
7.0215
7.0156
6.9453
6.9279
6.7369
6.7175
6.6959
4.6169
3.6106
3.5309
3.4269
1.00
5.17
4.16
4.01
2.19
3.14
1.95
3.11
2.12
2.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
N
N
O
Br
CO2Me
9c
NAME phr-7-23b
EXPNO 2
PROCNO 1
Date_ 20091110
Time_ 9.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700313 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


169.47
158.09
148.68
147.54
146.29
144.47
138.57
136.66
131.59
129.84
129.28
128.61
128.48
128.35
126.52
126.25
122.49
122.28
122.13
121.38
118.47
113.66
77.37
77.05
76.73
58.29
52.28
33.00
29.09
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
N
N
Br
O
CO2Me

9c


BRUKER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7.3725
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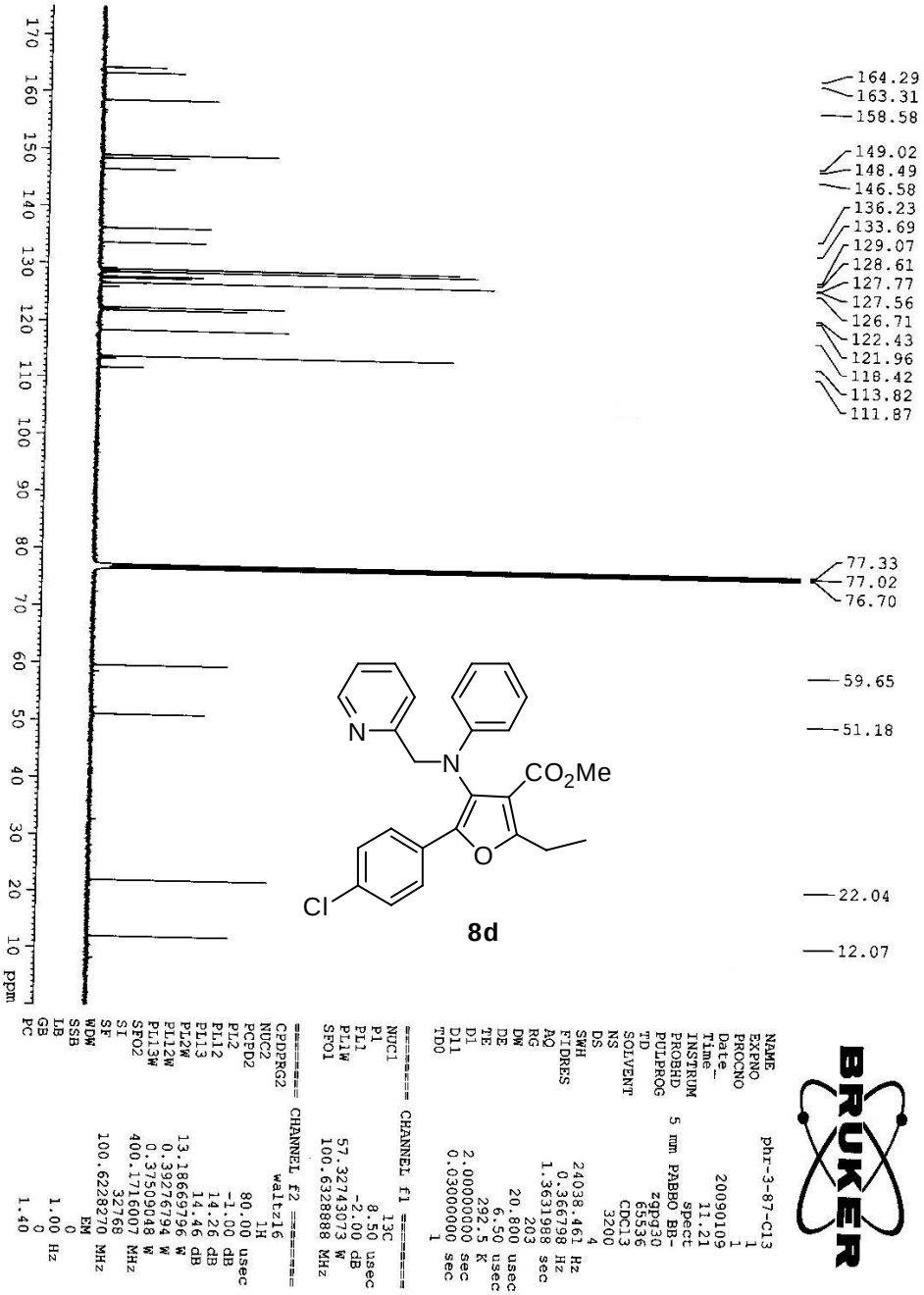
7.2820
7.2671
7.2628
7.2479
7.2436
7.0900
7.0822
7.0771
7.0652
7.0606
7.0545
7.0403
7.0343
7.0203
7.0002
6.9150
6.9017
6.8975
6.8849
6.6414
6.6267
6.6231
6.6061
4.6388
3.4447
2.9416
2.9227
2.9038
2.8850
1.1941
1.1752
1.1563
1.00
2.07
1.05
2.19
1.01
2.14
1.06
3.19
2.06
3.08
2.11
3.18
ppm
N
N
CO2Me
O
Cl
8d
BRUKER
NAME phr-4-33a
EXPNO 1
PROCNO 1
Date_ 20081230
Time 16.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 290.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700713 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00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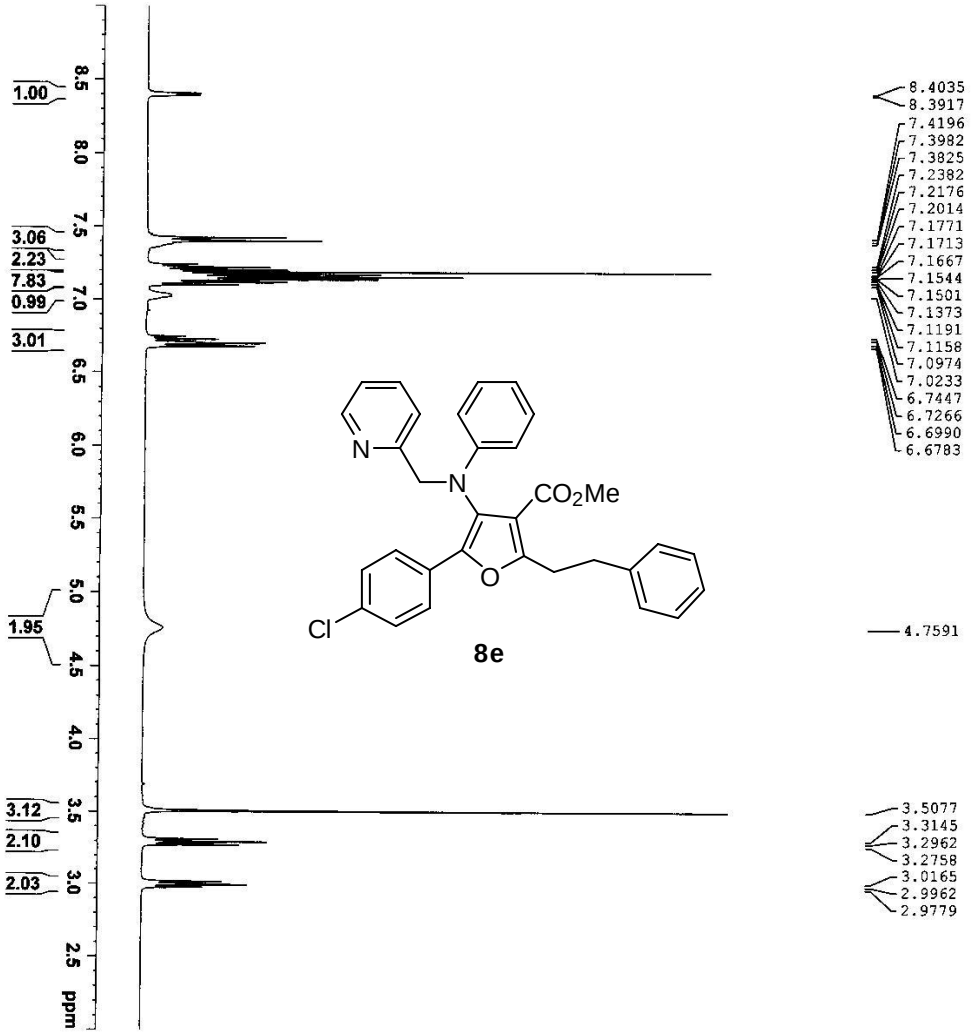
148.49
146.58
136.23
133.69
129.07
128.61
127.77
127.56
126.71
122.43
121.96
118.42
113.82
111.87
77.33
77.02
76.70
59.65
51.18
22.04
12.07
N
N
CO2Me
O
Cl
8d
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
NAME phr-3-87-C13
EXPNO 1
PROCNO 1
Date_ 20090109
Time_ 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 292.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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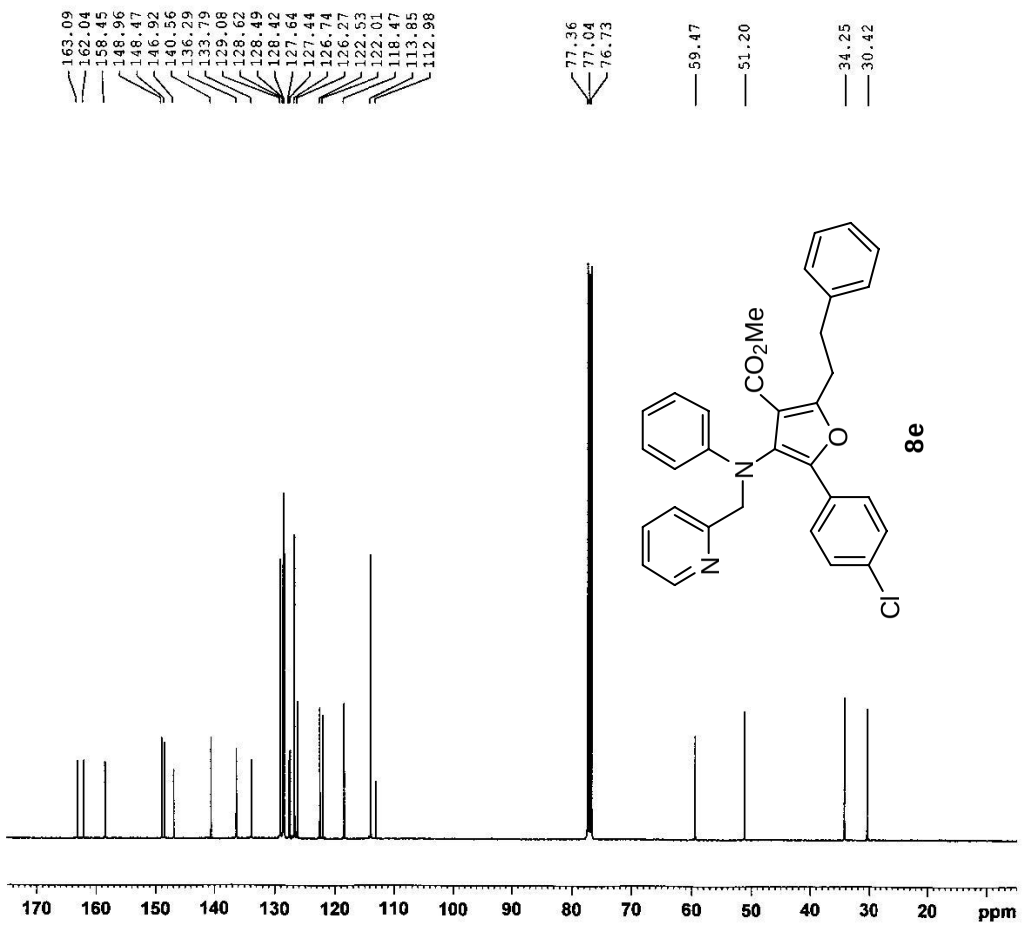
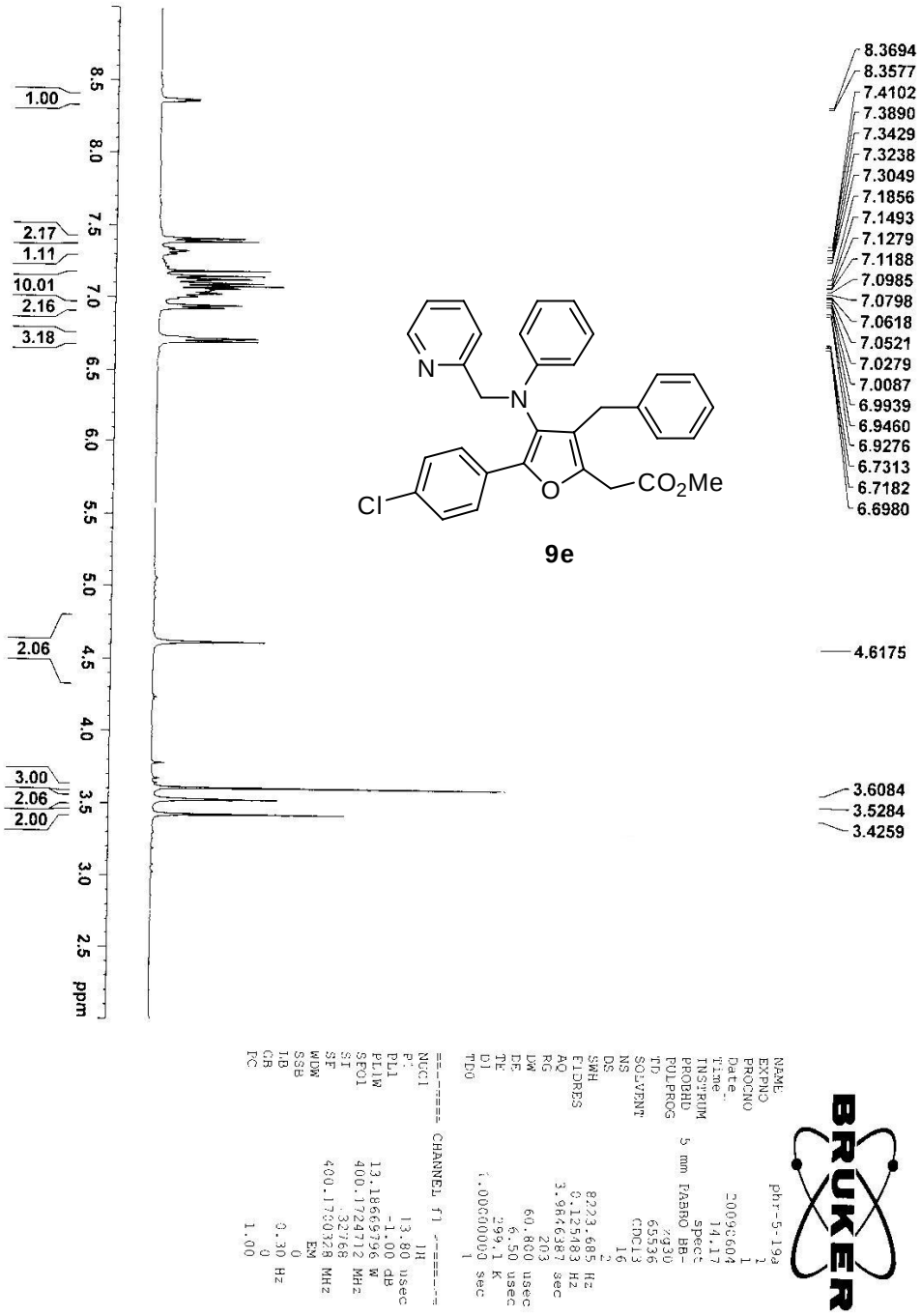
7.3825
7.2382
7.2176
7.2014
7.1771
7.1713
7.1667
7.1544
7.1501
7.1373
7.1191
7.1158
7.0974
7.0233
6.7447
6.7266
6.6990
6.6783
4.7591
3.5077
3.3145
3.2962
3.2758
3.0165
2.9962
2.9779
1.00
3.06
2.23
7.83
0.99
3.01
1.95
3.12
2.10
2.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
N
N
CO2Me
O
Cl
8e
NAME xj-2-31-a
EXPNO 1
PROCNO 1
Date_ 20080710
Time_ 17.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PLIW 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700320 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER



BRUKER

NAME	xj-2-31-a-C13	
EXPNO	1	
PROCNO	1	
Date_	20080710	
Time	17.51	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	6000	
DS	4	
SWH	24038.461	Hz
FIDRES	0.366798	Hz
AQ	1.3631988	sec
RG	203	
DW	20.800	usec
DE	6.50	usec
TE	298.8	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	8.50	usec
PL1	-2.00	dB
PL1W	57.32743073	W
SFO1	100.6328888	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	-1.00	dB
PL12	14.26	dB
PL13	14.46	dB
PL2W	13.18669796	W
PL12W	0.39276794	W
PL13W	0.37509048	W
SFO2	400.1716007	MHz
SI	32768	
SF	100.6228270	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	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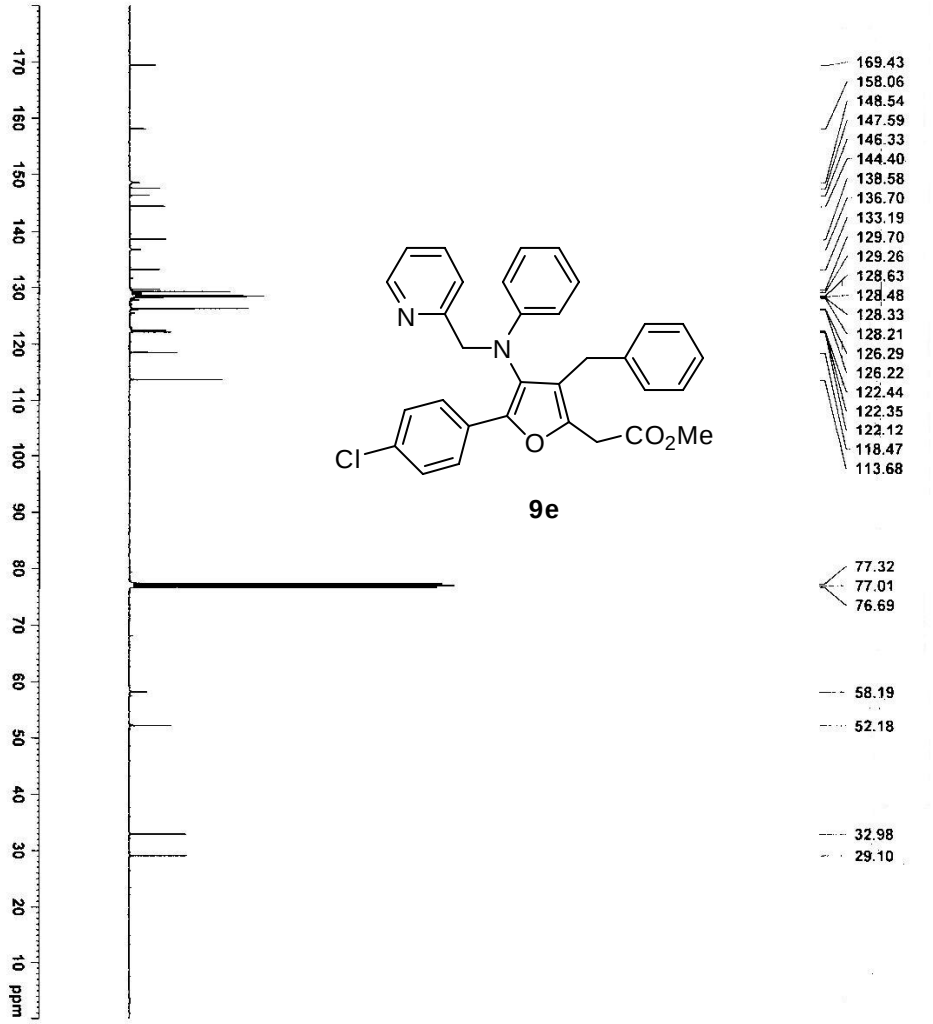
7.1493
7.1279
7.1188
7.0985
7.0798
7.0618
7.0521
7.0279
7.0087
6.9939
6.9460
6.9276
6.7313
6.7182
6.6980
4.6175
3.6084
3.5284
3.4259
1.00
2.17
1.11
10.01
2.16
3.18
2.06
3.00
2.06
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
N
N
Cl
O
CO2Me
9e
NAME phr-5-19a
EXPNO 1
PROCNO 1
Date_ 20090604
Time 14.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18666796 W
SFO1 400.1729712 MHz
SI 32768
SF 400.1732328 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00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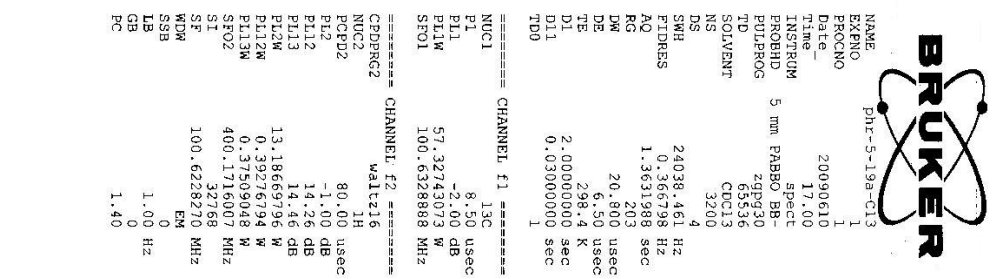
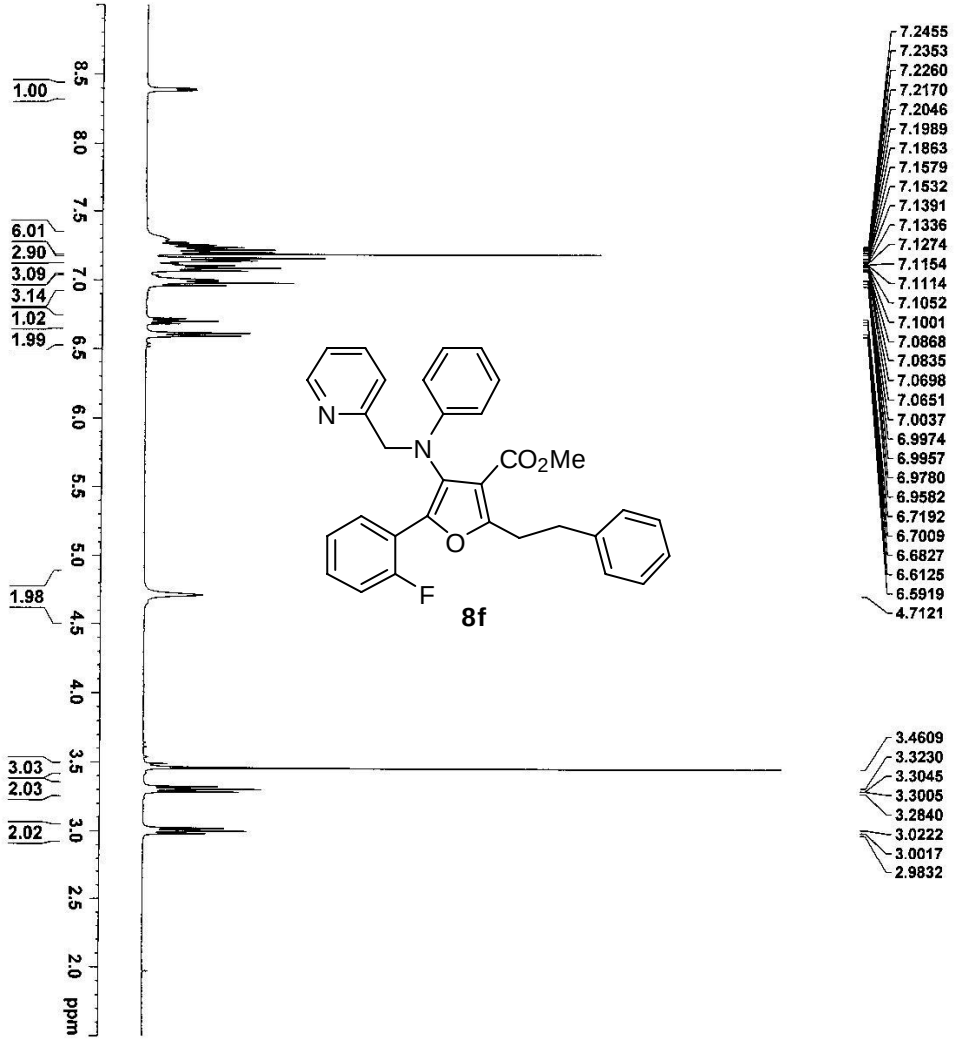
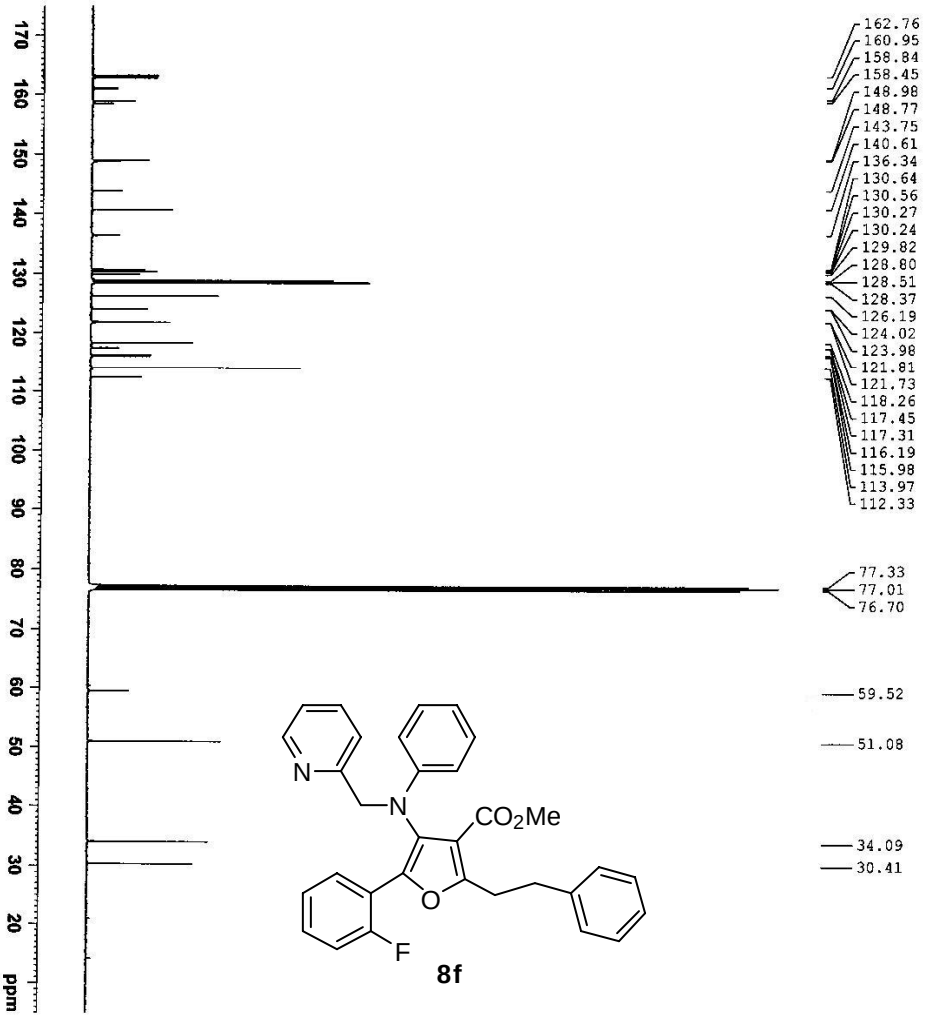
136.70
133.19
129.70
129.26
128.63
128.48
128.33
128.21
126.29
126.22
122.44
122.35
122.12
118.47
113.68
77.32
77.01
76.69
58.19
52.18
32.98
29.10
N
N
Cl
O
CO2Me
9e
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


BRUKER
NAME phr-5-19a-C13
EXPNO 1
PROCNO 1
Date_ 20090610
Time_ 17.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
NAME                  phr-XJ-2-10
EXPNO                          1
PROCNO                         1
Date_              20090915
Time_                  11.07
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                         65536
SOLVENT                  CDCl3
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.98463387 sec
RG                           203
DW                        60.800 usec
DE                         6.50 usec
TE                        300.1 K
D1                     1.00000000 sec
TD0                            1

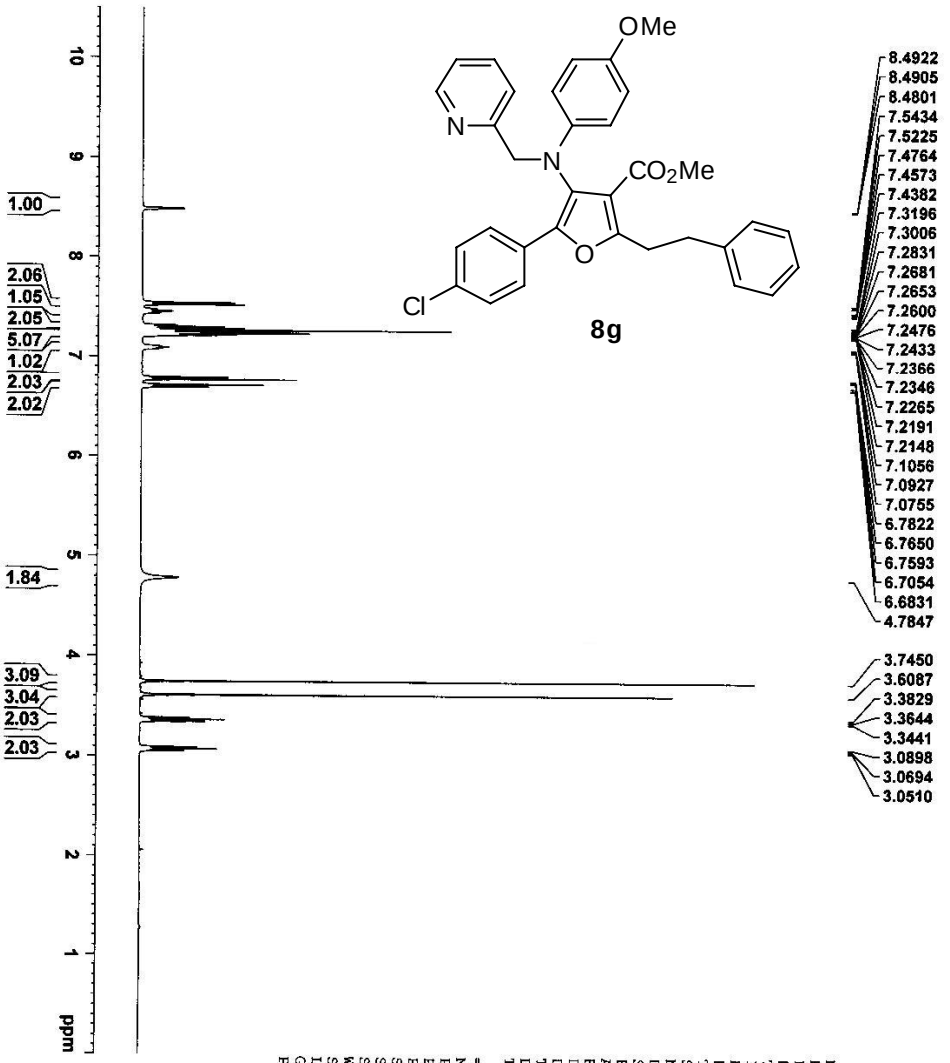
======== CHANNEL f1 ========
NUC1                         1H
P1                        14.20 usec
PL1                       -1.00 dB
PL1W                13.18669796 W
SFO1                400.1724712 MHz
SI                         32768
SF                  400.1700326 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



```
NAME                  PHR-Xj-C13
EXPNO                          1
PROCNO                         1
Date_                   20080715
Time_                      17.14
INSTRUM                     spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                           65536
SOLVENT                   CDCl3
NS                           12000
DS                             4
SWH                    24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                           203
DW                        20.800 usec
DE                         6.50 usec
TE                        298.8 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                          8.50 usec
PL1                        -2.00 dB
PL1W                57.32743073 W
SFO1               100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                       -1.00 dB
PL12                      14.26 dB
PL13                      14.46 dB
PL2W                13.18669796 W
PL12W                0.39276794 W
PL13W                0.37509048 W
SFO2               400.1716007 MHz
SI                          32768
SF                 100.6228270 MHz
WDW                            EM
SSB                             0
LB                         1.00 Hz
GB                              0
PC                         1.40
```


OMe
N
N
CO2Me
O
Cl
8g
8.4922
8.4905
8.4801
7.5434
7.5225
7.4764
7.4573
7.4382
7.3196
7.3006
7.2831
7.2681
7.2653
7.2600
7.2476
7.2433
7.2366
7.2346
7.2265
7.2191
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7.0927
7.0755
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6.7650
6.7593
6.7054
6.6831
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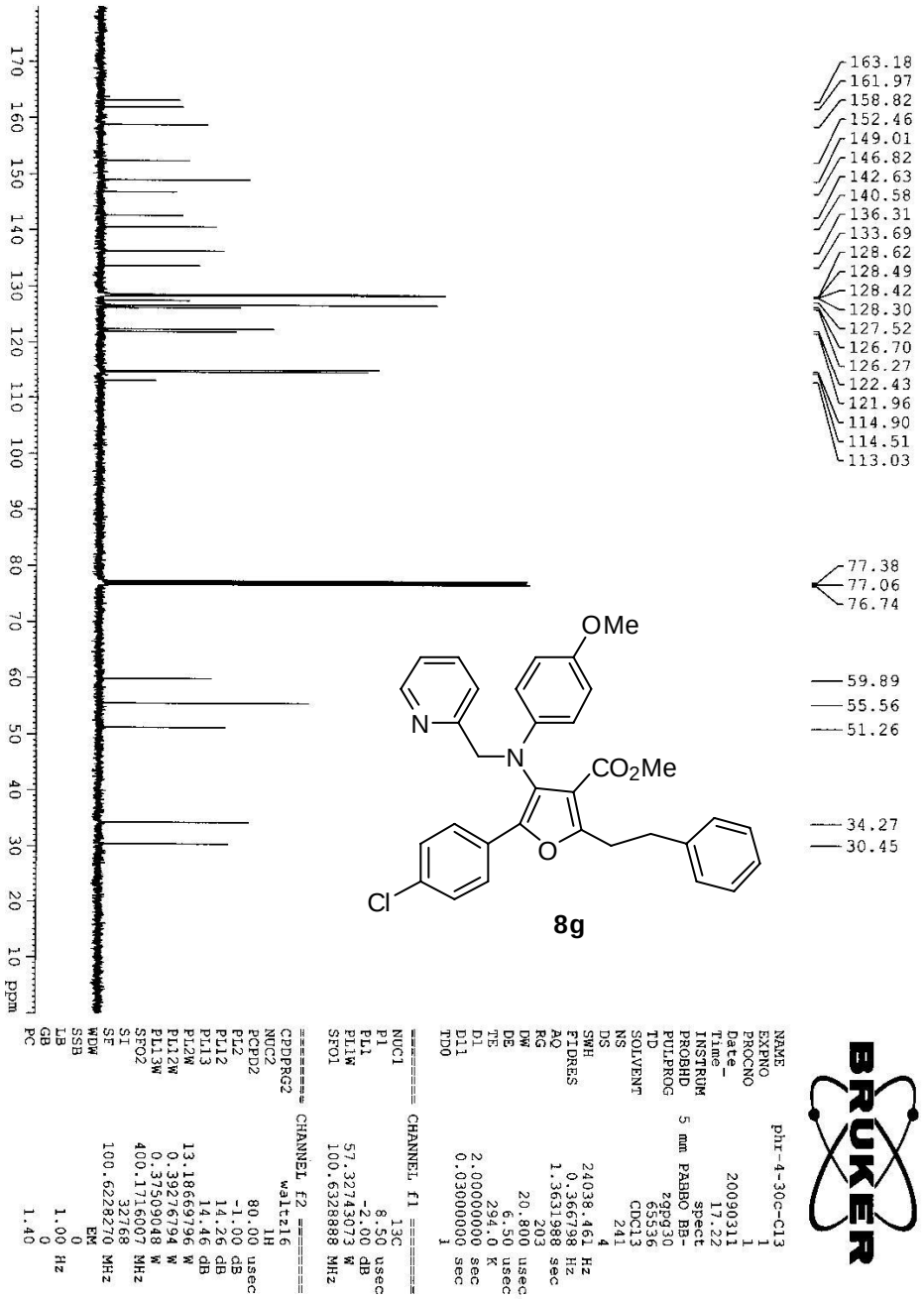
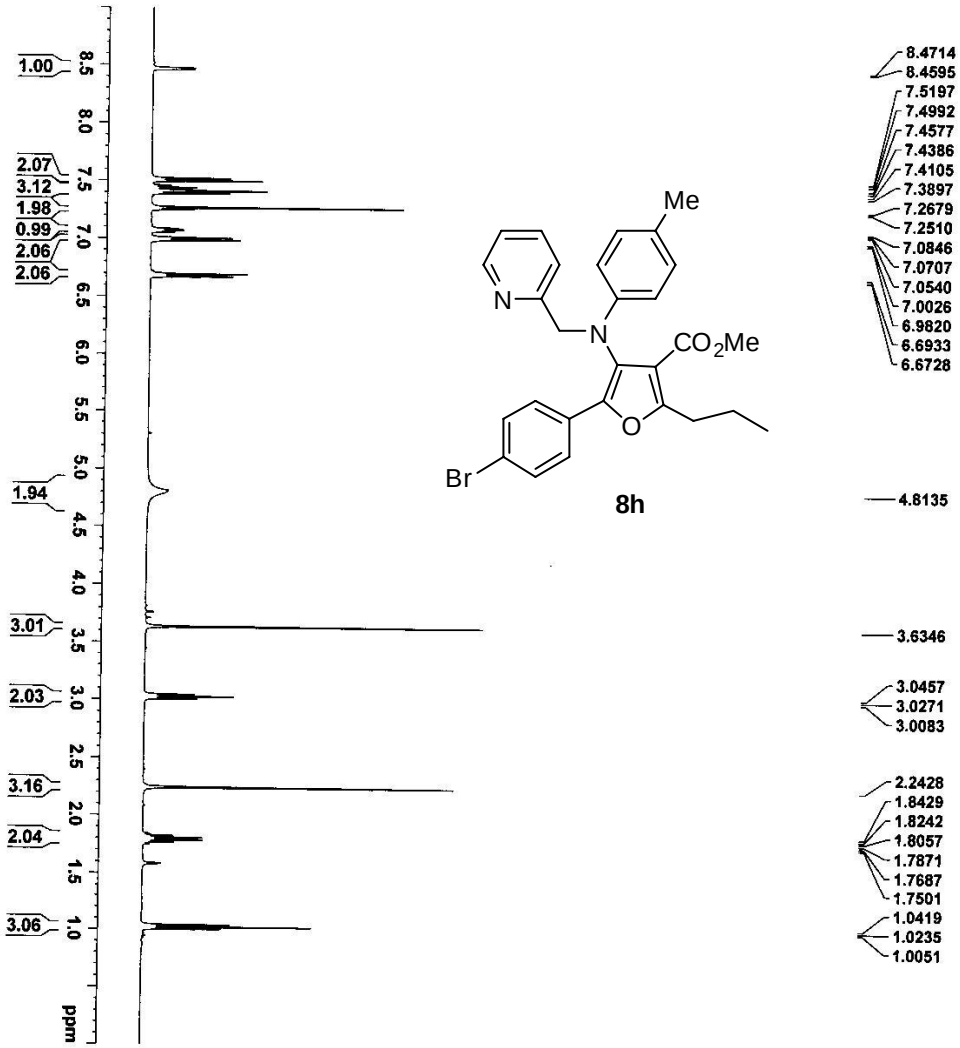
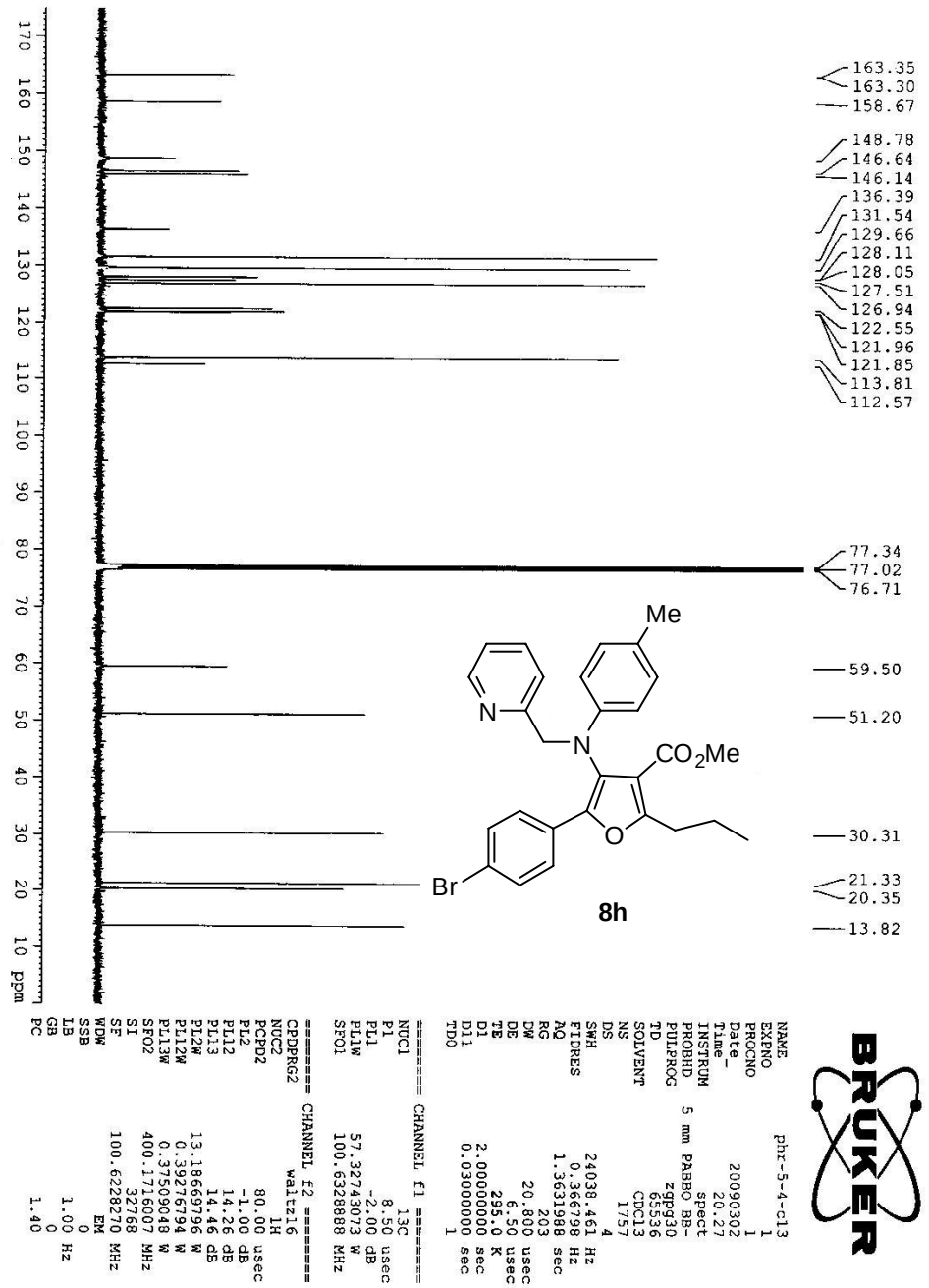
3.6087
3.3829
3.3644
3.3441
3.0898
3.0694
3.0510
1.00
2.06
1.05
2.05
5.07
1.02
2.03
2.02
1.84
3.09
3.04
2.03
2.03
ppm
NAME phr-4-30c
EXPNO 1
PROCNO 1
Date_ 20081230
Time_ 15.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 291.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
BRUKER


163.18
161.97
158.82
152.46
149.01
146.82
142.63
140.58
136.31
133.69
128.62
128.49
128.42
128.30
127.52
126.70
126.27
122.43
121.96
114.90
114.51
113.03
77.38
77.06
76.74
59.89
55.56
51.26
34.27
30.45
OMe
N
N
CO2Me
O
Cl
8g
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
NAME phr-4-30c-C13
EXPNO 1
PROCNO 1
Date_ 20090311
Time_ 17.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 241
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.4714
8.4595
7.5197
7.4992
7.4577
7.4386
7.4105
7.3897
7.2679
7.2510
7.0846
7.0707
7.0540
7.0026
6.9820
6.6933
6.6728
4.8135
3.6346
3.0457
3.0271
3.0083
2.2428
1.8429
1.8242
1.8057
1.7871
1.7687
1.7501
1.0419
1.0235
1.0051
1.00
2.07
3.12
1.98
0.99
2.06
2.06
1.94
3.01
2.03
3.16
2.04
3.06
ppm
Me
N
N
CO2Me
O
Br
8h
NAME phr-5-4
EXPNO 1
PROCNO 1
Date_ 20090224
Time 16.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00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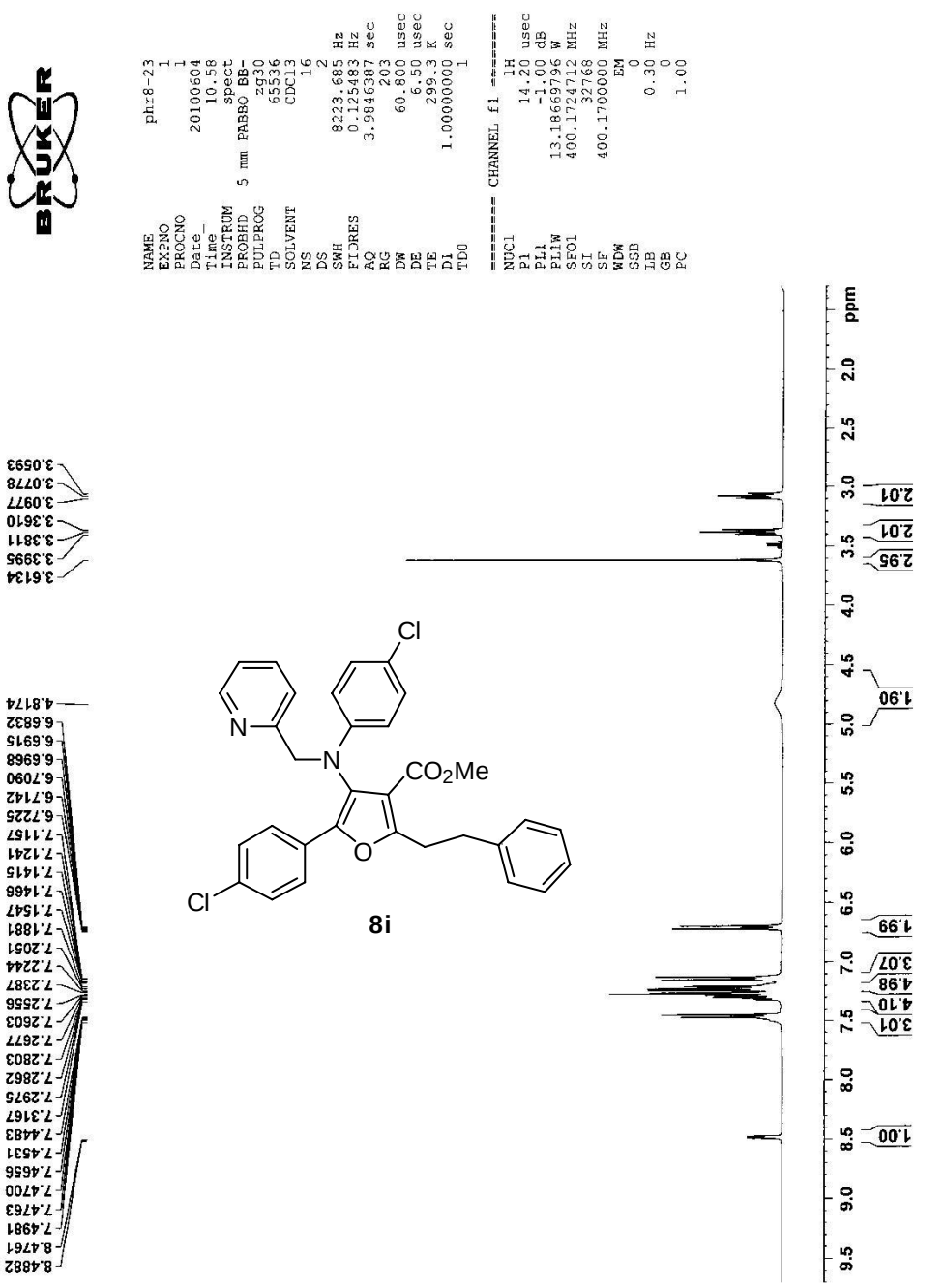
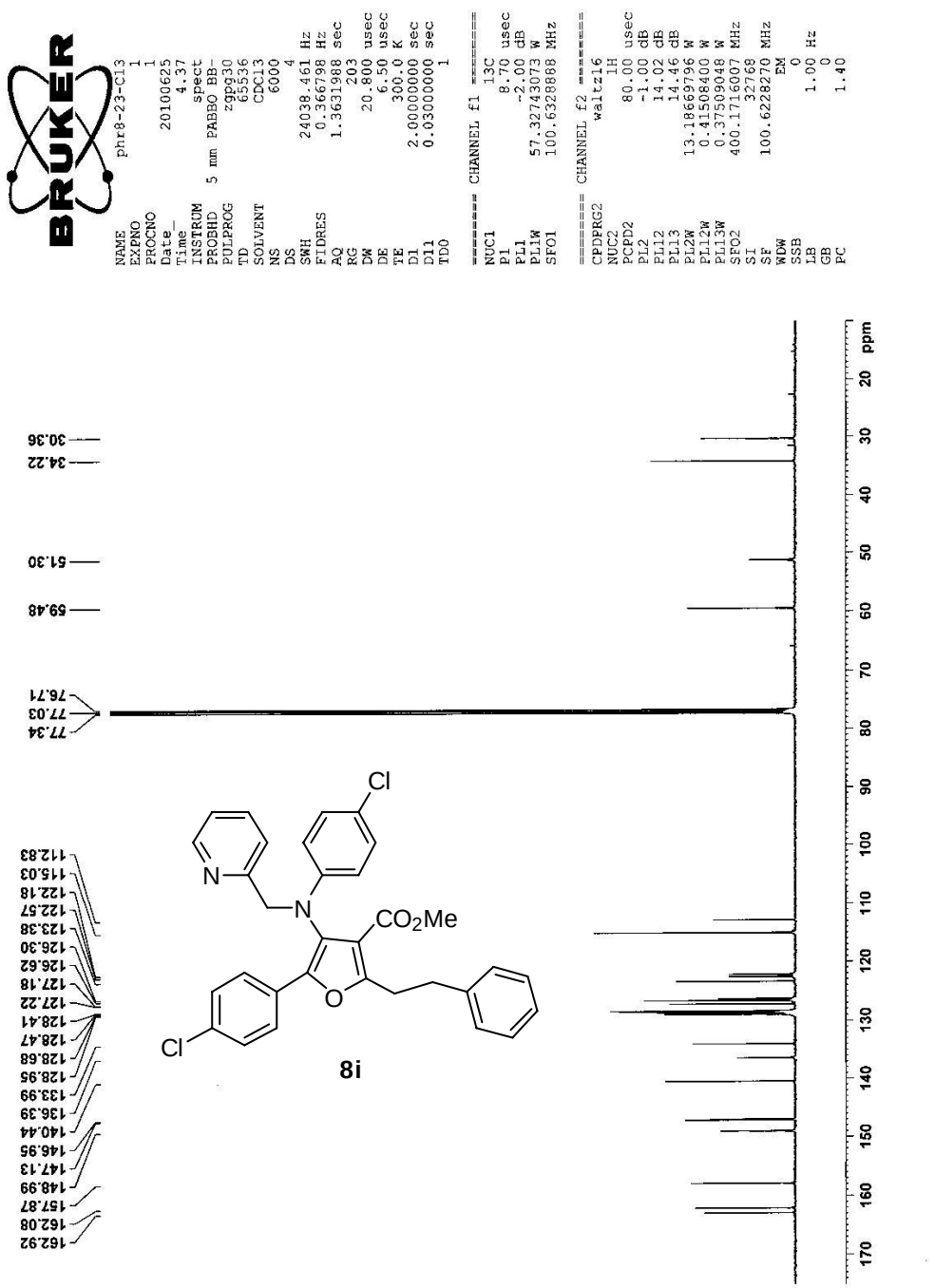
163.30
158.67
148.78
146.64
146.14
136.39
131.54
129.66
128.11
128.05
127.51
126.94
122.55
121.96
121.85
113.81
112.57
77.34
77.02
76.71
59.50
51.20
30.31
21.33
20.35
13.82
Me
N
N
CO2Me
O
Br
8h
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
NAME phr-5-4-c13
EXPNO 1
PROCNO 1
Date_ 20090302
Time 20.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1757
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


NAME phr8-23
EXPNO 1
PROCNO 1
Date_ 20100604
Time_ 10.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
N
N
CO2Me
O
Cl
8i
ppm
2.01
2.01
2.95
1.90
1.99
3.07
4.98
4.10
3.01
1.00


NAME phr8-23-c13
EXPNO 1
PROCNO 1
Date_ 20100625
Time_ 4.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328868 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.02 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.41508400 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
30.36
34.22
51.30
59.48
76.71
77.03
77.34
112.83
115.03
122.18
122.57
123.38
126.30
126.62
127.18
127.22
128.41
128.47
128.68
128.95
133.99
136.39
140.44
146.95
147.13
148.99
157.87
162.08
162.92
Cl
N
N
CO2Me
O
Cl
8i
ppm